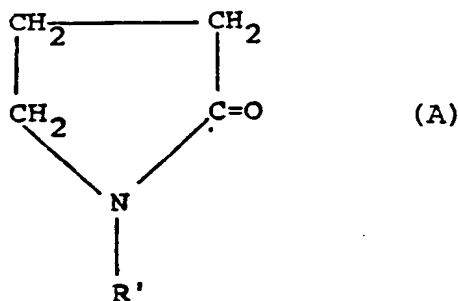




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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification⁴ : C07D 207/267, 207/27, 211/76 C07D 223/10	A1	(11) International Publication Number: WO 88/ 00184 (43) International Publication Date: 14 January 1988 (14.01.88)
(21) International Application Number: PCT/US87/01391 (22) International Filing Date: 16 June 1987 (16.06.87) (31) Priority Application Numbers: 879,776 013,760 (32) Priority Dates: 27 June 1986 (27.06.86) 12 February 1987 (12.02.87) (33) Priority Country: US (71) Applicant: GAF CORPORATION [US/US]; 1361 Alps Road, Wayne, NJ 07470 (US). (72) Inventors: LOGIN, Robert, B. ; 137 Page Drive, Oakland, NJ 07436 (US). CHAUDHURI, Ratan, K. ; 74 Reservoir Avenue, Butler, NJ 07405 (US). HASHEM, Mohamed, M. ; 100 Magnolia Place, Wayne, NJ 07470 (US). HELIOFF, Michael, W. ; 485 Otisco Drive, Westfield, NJ 07090 (US). PRITCHARD, David, W. ; 74 Cedar Ridge Drive, Vernon, NJ 07462 (US). RUPPERT, Ronald, M. ; 8 Sova Place,		Moonachie, NJ 07074 (US). SAVIO, Lenore, E. ; 188 Buckingham Way, Somerset, NJ 08873 (US). SUWALA, David, W. ; 81 Chatham Ct., Dover, DE 19901 (US). TRACY, David, J. ; 209 Comly Road, Lincoln Park, NJ 07035 (US). (74) Agents: MAUE, Marilyn, J. et al.; GAF Corporation, 1361 Alps Road, Building # 10, Patent Dept., Wayne, NJ 07470 (US). (81) Designated States: AT (European patent), AU, BE (European patent), CH (European patent), DE (European patent), DK, FR (European patent), GB (European patent), IT (European patent), JP, LU (European patent), NL (European patent), NO, SE (European patent). Published <i>With international search report.</i> <i>Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>

(54) Title: SURFACE ACTIVE LACTAMS**(57) Abstract**

N-hydrocarbon substituted lactams, particularly N-alkyl substituted lactams having formula (A), wherein R' is a hydrophobic radical such as linear or branched chain alkyl group containing from 8 to 20 carbon atoms, most preferably 8 to 14 carbon atoms; which lactams are capable of forming micelles in aqueous solutions irrespective of pH and have critical micelle concentrations of between about 1×10^{-3} and about 1×10^{-5} moles per liter. The invention also relates to the uses of the N-hydrocarbon substituted lactams which involve surfactant properties, such as solubility, wetting, viscosity building, emulsifying, foam boosting and/or complexing properties.

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SURFACE ACTIVE LACTAMSRELATED CASE

This is a continuation-in-part of co-pending application Serial No. 879,776, filed June 27, 1986, entitled "SURFACE ACTIVE LACTAMS".

BACKGROUND OF INVENTION

In one aspect this invention relates to a particular group of N-hydrocarbon substituted lactams having novel properties. In another aspect the invention relates to utilization of such lactams in specific fields of application where said properties are required to provide significant advantages.

N-lower alkyl pyrrolidones have found wide commercial acceptance as non-toxic aprotic chemical solvents. However, absence of hydrophobic-lipophobic balance in these molecules, as in the case of N-methyl pyrrolidone, prevents micellular formation; consequently they possess no significant aqueous surfactant properties. Amine oxides are known to possess high surfactant activity; however these compounds are not stable at high temperatures and cannot be employed in metal working or high temperature fiber processing.

Accordingly, it is an object of the present invention to overcome the above deficiencies by providing improved surfactant-complexant agents which are readily obtained in an inexpensive and commercially feasible manner.

Another object of this invention is to provide a group of compounds having excellent surfactant and complexing properties.

Another object is to provide a group of compounds having viscosity building, wetting, and/or anti-corrosive properties.

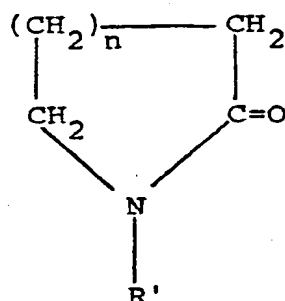
Still another object is to provide specialized uses for a group of compounds having the above properties.

These and other objects of the invention will become apparent from the following description and disclosure.

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THE INVENTION

According to this invention there is provided a distinctive group of N-hydrocarbon substituted lactams having unusual properties and defined by the formula



A.

wherein n is an integer having a value of from 1 to 3 and R' is a hydrophobic radical selected from the group consisting of a linear, branched chain or cyclic alkyl radical containing from 7 to 22 carbon atoms; a naphthyl or alkyl substituted naphthyl radical containing from 10 to 26 carbon atoms and an alkylphenyl or phenylalkyl radical containing from 9 to 26 carbon atoms; which lactams are capable of forming micelles in neutral, basic or acidic aqueous media or have a critical micelle concentration of between about 1×10^{-3} and about 5×10^{-5} moles per liter. Of the above lactam surfactants, the N-alkyl lactams are preferred and, of these, N-alkyl pyrrolidones containing 8 to 18 carbon atoms and having a critical micelle concentration less than 2×10^{-2} moles per liter are most preferred.

While it is intended to include lactams substituted with cycloalkyl R' groups, it is found that the hydrophobic affect of these cyclic groups is less than that of their linear analogs. Accordingly, where a N-cycloalkyl R' group is intended, the R' moiety contains at least 8 carbon atoms to provide hydrophobic-lipophobic balance.

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It is also intended to include lactams wherein one of the hydrogen atoms bonded to a carbon atom member of the heterocyclic ring can be substituted with methyl or ethyl.

5 The cyclic hydrophilic moiety of the compounds herein defined is an important factor in maximizing their efficiency by concentrating the molecule, and thus the surfactant activity, at the interface and minimizing solubility of the molecule in liquid phases.

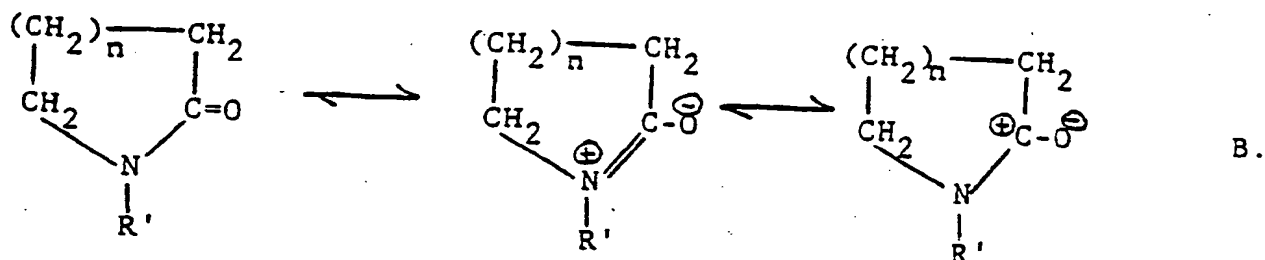
10 The above properties demonstrate the high efficiency and activity of the present nonionic surfactants. Such surfactant properties are unexpected since the compounds lack the polyalkoxy groups commonly associated with conventional nonionic surfactants. By way of comparison, N-dodecyl-2-pyrrolidone, exhibits a surfactant properties equivalent to a lauryl alcohol containing five moles of ethoxylate, i.e. $C_{12}H_{25}-(CH_2CH_2O)_5H$, a known commercial nonionic surfactant.

20 Not all of the present lactams possess good aqueous surfactant properties. For example, the pyrrolidones having R' substituents containing less than 8 carbon atoms do not have hydrophobic groups of a length sufficient to form stable micelles in water. On the other hand, R' alkyl substituents of hexadecyl and above exhibit an imbalance such that the hydrophilic pyrrolidone or caprolactam moiety cannot counteract the dominating hydrophobic character of the alkyl group. However, these higher molecular weight compounds can provide surfactant properties in non-aqueous systems and are valuable wetting agents in applications involving solid substrates.

30 The structure of the present lactams provides a key to their unique properties. Specifically, the highly polar and hydrophilic pyrrolidone moiety exhibits several resonance forms, i.e.

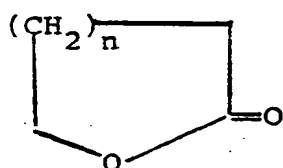
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The existence of such multiple resonance states contributes to the high dipole moment of about 4 debye, possessed by these lactams. A high dipole moment is an important property, conferring the ability of these molecules to assume multiple resonance forms which promotes their tendency to complex or interact with many chemical groups. For example, the ability of these compounds to interact with anionic moieties, e.g. sulfate groups, is beneficial in enhancing foam and reducing skin irritability of various medicinal creams and lotions containing such or similar anionic groups.

The above lactam products having a molecular weight of from about 180 to about 450 are conveniently prepared by several known processes including the reaction between a lactone having the formula



wherein n is as defined above, and an amine having the formula $R'-NH_2$ wherein R' is as defined above. The amine reactant having the formula $R'-NH_2$ includes alkylamines having from 7 to 20 carbon atoms; naphthyl or alkyl naphthyl amines having from 10 to 26 carbon atoms; alkylphenyl or phenylalkyl amines having from 9 to 26 carbon atoms; amines derived from natural products, such

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as coconut amines or tallow amines and distilled cuts or hydrogenated derivatives of such fatty amines. Also, mixtures of amine reactants can be used in the process for preparing the present compounds. Such mixtures can include cyclic, linear and branched chain amino species having an alkyl or other organic substituent of the same or different molecular weight. In the present process the amine and lactone reactants, combined in a mole ratio of from about 1:1 to about 1:5, are reacted under conditions of constant agitation, at a temperature between about 200°C. and about 350°C. under a pressure of from atmospheric to about 650 psig for a period of from about 1 to about 15 hours; preferably at 250°C. to 300°C. under an initial ambient pressure for a period of from 5 to 10 hours. The resulting lactam product is recovered and purified by distillation or by any other convenient recovery process.

The N-alkyl lactam products having 11 to 14 carbon atoms are clear, water white liquids, at room temperature; whereas those having 16 or more carbon atoms are solids. These lactams have a neutral or slightly basic pH, a surface tension between about 25 and about 35 dynes/cm as a 0.1% water solution and a viscosity of from about 6 to about 30 cps at 25°C. More particularly, these lactams of 97% or higher purity are capable of producing micelles at a critical molar micelle concentration less than 1×10^{-3} . For the N-alkyl-2-pyrrolidones species the capability of producing micelles at a critical micelle concentration of between about 4.4×10^{-4} and about 5×10^{-5} is obtainable. Generally, the C_8 to C_{14} alkyl lactams display primarily surfactant properties; whereas the C_{16} to C_{22} alkyl species are primarily complexing agents; although some degree of surfactants and complexing capability exists in all of the present species.

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The maximum surface concentration of the present surfactants at equilibrium above the critical micelle concentration is exceptionally high, i.e. between about 2 moles/cm x 10⁻¹⁰ and about 5 moles/cm x 10⁻¹⁰. It is believed that the monodisperse nature of the hydrophobe and hydrophile moieties of these surfactants contribute to the high surface concentrations by maximizing entropy of packing, particularly in the case of the N-decyl-2-pyrrolidone species which exhibits essentially equal hydrophobe/hydrophobe and hydrophile/hydrophile interactions at the liquid interface, while longer R groups reduce packing due to steric effects.

The present group of lactams also exhibit a unique and heretofore unrealized combination of properties, suitable for increasing rates of solubilization for solids in a liquid medium in which they are normally insoluble.

Many drugs having little or no solubility in water are orally administered in the form of pills, tablets or capsules. Examples of such drugs include hydrochlorotriazide, chlorothiazide, griseofulvin, progesterone, phenyl butazone, and sulfathiazole. These drugs may be formulated with non-pyrrolidonic anionic surfactants such as sodium lauryl sulfate, dioctyl sodium sulfosuccinate or non-ionic surfactants such as polyoxyethylene (20) sorbitan monooleate (Tween 80) or polyoxyethylene (23) lauryl ether. Polyoxyethylene based non-ionic surfactants may contain dioxane and, in the case of those derived from fatty amines such as polyoxyethylated (5) tallow amine, nitrosoamines which are well known carcinogens. The present lactams, besides their ability to diminish aggregation of drug particles and increasing the area available for dissolution, are free of dioxane and nitrosoamine contaminants and the

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5 purity of these non-alkoxylated compounds is more readily assured since they are not subject to variations of extraneous by-product contamination or degree of alkylene oxide polymerization. Unlike sodium lauryl sulfate, the present pyrrolidones are non-irritating and do not react with acidic drugs or pharmaceutical excipients.

10 Additionally, the present lactams possess the ability to solubilize a drug in its micelles, as well as the capability of lowering surface tension to permit water and digestive fluid penetration into the drug mass, thus increasing the rate of drug entry into the blood stream. In certain cases, where dissolution is the rate-limiting step in the absorption process, these lactams in many instances actually increase the normal peak saturation levels in the blood for a particular drug.

15 A particularly beneficial property of instant lactams is their complexing ability in which the complexed active compound is positioned on the micelle surface. The lactam containing molecules in turn migrate to the surface of a bulk solution forming a monomolecular film thereon; thus concentrating the chemical which has been complexed at the solution-substrate interface. This spacial concentration of the active ingredient at the liquid surface provides more efficient contact between the complexed chemical and the substrate, thus permitting the efficient use of an active ingredient to preform its intended function. The lower concentration of an active ingredient such as a drug having toxic properties, can result in lowering toxicity and alleviating undesirable side effects.

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The complexing capability of the present products involves a wide range of organic and inorganic compounds and includes compounds containing a phenolic group, a mercapto group, an acidic hydrogen, metal oxides compounds, metal salts, halogens and polarizable compounds. More specifically, these compounds include mineral acids such as the hydrogen halides, particularly hydrochloric acid, sulfuric, nitric, phosphoric acids, etc., organic acids such as chloroacetic, acetic, formic, etc., phenolics such as vanillin, phenol, resorcinol, etc. metal salts such as SnI_2 , $\text{BiC}_6\text{H}_5\text{O}_7$, SbF_3 , etc. and polarizable compounds such as I_2 , HI_3 , and resonant structures i.e. dyestuffs and fluorescent dyes and perfumary materials, etc. They also complex with urea and urea derivatives and odor forming components of human perspiration to mask or remove odor and stains caused by these components.

Additionally, they control objectionable odors emanating from metal treating and slaughter house operations as well as household odors on rugs, furniture, clothing, or encountered in pet environments, veterinary sites. The present surfactants also complex with odor forming bodies in animal and human wastes containing eg. mercaptans, urea, tars, nicotine, molds, and other odor causing chemicals.

The present N-alkylpyrrolidones are excellent clarifying agents for beverages, particularly beer and wines, which contain phenolic and other impurities. Removal of contaminants results in a product having improved clarity, taste and color. Beverage impurities can be removed by several methods. For example, water soluble pyrrolidones of this invention, also soluble in the beverage, are complexed with the phenolic impurities at a temperature below their respective cloud points.

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When the beverage is heated above the cloud point of the lactam employed, the resulting complexed impurities, eg. polyphenols/lactam, are separated and thus removed. This method of clarification is beneficially employed using N-alkyl C₁₀ to C₁₄ pyrrolidones. Water insoluble N-alkyl lactams of this invention e.g. N-hexadecyl, N-octadecyl and N-tallow pyrrolidones are also useful and are employed to extract, impurities by intimately mixing the lactam and beverage. The phenolic impurities, mainly polyphenols, preferentially complex with the heterocyclic ring of the water insoluble lactam and are then removed by filtration.

Still further the complexability and high heat stability of the present lactams makes them valuable decontaminating agents for industrial wastes including radio active waste material. For example radioactive halogens complex with certain of the present lactams to provide a less toxic water solubule product which can be washed off vegetation on which animals feed; also water insoluble lactams can be used to complex with and extract radioactive material from aqueous solutions, thereby concentrating them in a less toxic form which can be more economically dealt with. Further, complexed radioactive products can be used as liquid feed to a fixation process which converts the product into a solid non-leachable material for permanent storage, as in the processes described in U.S. patents 3,206,409; 3,153,566; 3,213,031 and 3,110,557. With increasing use of nuclear reactors for power and production, it is becoming increasingly urgent to devise safer methods for disposal of fission by-products or waste materials in a less toxic form. The present lactams can also be used in the purification of water, e.g. a sewage treatment, to remove organic pollutants from aqueous streams by an economical low energy process.

- 10 -

Still another benefit obtained by the complexing capability of the present compounds is realized by their ability to reduce toxicity of those systemic and non-systemic chemicals whose toxicity levels exceed the use intended. Pesticides, insecticides, fungicides and herbicides all contain members in this category. For example, 2-methyl-2-(methythio) propionaldehyde O-(methyl carbamoyl) oxime, known as Aldicarb, when mixed with water produces a ecologically hazardous mixture. However, complexing this compound with one of the present compounds reduces its toxicity to a level sufficient to effectively kill household insects without danger to humans. The toxicity of phenyl mercuric compounds such as the acetate, borate, chloride, hydroxide, nitrate, naphthenate, oleate, propionate and salicylate as well as that of aldrin and dieldrin are significantly reduced through complexing. Other herbicides and pesticides having normal required toxicity can be incorporated with the lactams of this invention to provide better adhesion on the surface of plant tissue, increased persistence of chemical action, reduced interfacial tension between the active component and plant surface and other benefits commensurate with the particular properties of the lactam selected. Also, because the present compounds tend to concentrate the active ingredient at the surface of its micellular structure, smaller dosages of the active ingredient can be employed and more efficiently utilized. Accordingly, the present lactams serve as valuable adjuvants for active ingredients in liquid solution which are applied to a substrate.

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The performance of non-systemic active ingredients depends on the quality of the spray deposit. Particularly fungicides require a complete and uniform coverage which is resistant to heavy dews, rainfall and sprinkler irrigation. N-n-dodecyl-2-propylene promoted polyvinylpyrrolidone, most preferably in about an 80/20 wt % mixture with an alkyd-resin based compound is particularly beneficial in providing good sticking performance on crops with a wide variety of active ingredients. Application levels of from about 30 to about 130 ml/100 liters of spray, depending on total spray volume, type of crop and active ingredient, are suitable.

Also, superior oil/surfactant blends are formed, e.g. by 85/15 wt % mixtures of N-octyl-2-pyrrolidone and N-dodecyl-2-pyrrolidone, with a non-phytotoxic paraffin oil. Such blends are more effective than conventional types of spray oils because of the synergistic effect between the spray oil and the surfactant blend.

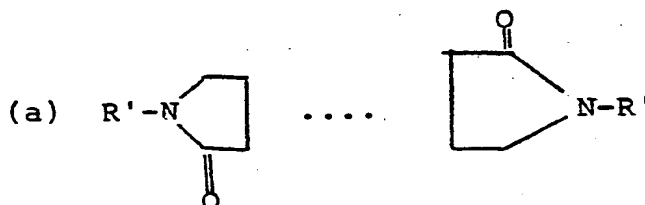
Another remarkable and unexpected property of the N-alkyl pyrrolidones of this invention is the reverse solubility exhibited by the C₈ and C₁₄ alkyl species, hereinafter discussed in more detail.

The cloud points of 10% aqueous solutions of the following compounds are reported for various N-alkyl-2-pyrrolidones

<u>alkyl group</u>	<u>Cloud Point</u>
cyclohexyl	55°C.
octyl	< 0°C.
decyl	19°C.
dodecyl	15-19°C.
tetradecyl	33°C.
coco	22-23°C.
hexadecyl	insoluble

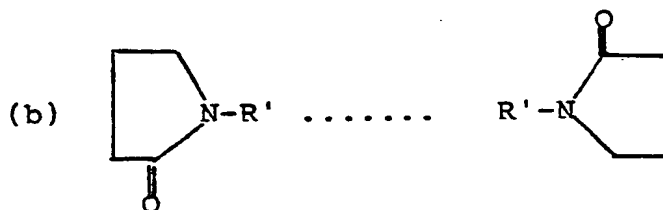
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The cloud points indicate the ability of the lactam molecule to retain water of hydration. The higher the cloud point, the more soluble, i.e. hydrated, the surfactant. As shown above, the cyclohexyl pyrrolidone species is highly soluble, requiring 55°C. to effect dehydration. However, this species fails to form stable micelles. Conversely, the octyl pyrrolidone species has a cloud point below room temperature and is almost insoluble. It appears that the octyl group has insufficient length to counteract the action of the pyrrolidone groups in the interactions (a) and (b)



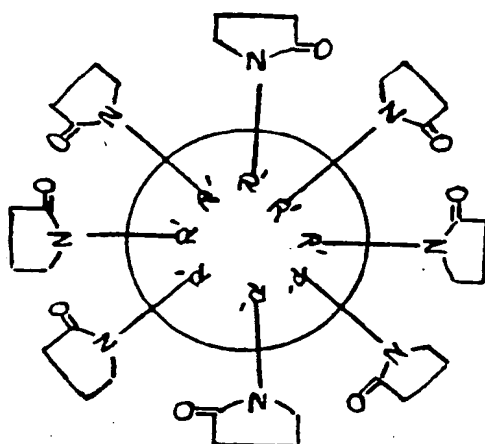
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For the octyl species, it is believed that interaction (a) predominates and the resulting dimer is insoluble for the reason that the R' groups are exposed in terminal positions of the interacted molecules thus reducing the hydration of the pyrrolidone moieties. Conversely, for longer chain R' groups, interaction (b) predominates and stable micelles i.e.



E.

where interaction occurs among the R' groups, are then formed.

The above properties combine to provide products of the present invention which are useful (1) in the solubilization of insoluble compounds such as for example insoluble drugs, cosmetics and agricultural chemicals to form surface active compositions, (2) as slip and antiblock agents for incorporation of components into plastic films to decrease the coefficient of friction, (3) as hydrolysis resistant solvents and auxiliaries for use in either acid or caustic environments, (4) as paint or resin strippers in electronic applications, e.g. printed circuit boards, (5) in the separation of molecules, e.g. the separation of biologically active or other active molecules from fermentation broths or other

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solutions, (6) as viscosity builders to form gels and
pastes, (7) as a water-in-oil or oil-in-water emulsifiers
for cosmetics, particularly in hair and skin care
formulations (8) as antistatic agents (9) as beverage
clarifiers, particularly beer and wine clarifiers, (10) as
stain and spot removers for textiles, (11) as surfactants
for wettable powders and detergents, (12) as foam
stabilizers, (13) as agents for increasing the rate of
dissolution of normally insoluble tablets or pills, (14)
as detoxifying agents and many other uses which will
become apparent from the present description and
disclosure.

The lactams of this invention are soluble in most
organic solvents including glycerin, acetone, lower
molecular weight alcohols, toluene, polyethylene glycols,
polypropylene glycols, xylene, heptane, methylene
chloride, perhalogenated lower molecular weight
hydrocarbons, paraffin oil, Stoddard solvent, etc., and
are compatible with all classes of surfactants under
varying pH conditons. The water dispersibility properties
of the C_{10} to C_{14} alkyl species seems to contravert
the generally accepted chemical behavior of hydrocarbon
compounds. One would expect that as the alkyl chain
length increases, the compound would become more
hydrophobic. Instead, it is now discovered that while
N-octyl pyrrolidone is substantially insoluble in water,
the N-decyl species shows partial solubility and the
N-dodecyl and N-tetradecyl pyrrolidones are completely
water dispersable. Water insolubility is again evidenced
in the pyrrolidones having alkyl group of 16 or more
carbon atoms, for the reasons explained above. However,
blends of higher alkyl species with the C_8 to C_{14}
alkyl species are water dispersible by algebraic summation
of the individual contributions of the respective alkyl

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and pyrrolidone moieties to the hydrophobic/hydrophilic balance. Also a hydrotrope or a mineral acid electrolyte e.g., a hydrogen halide such as HCl, sulfuric acid, phosphoric acid, xylene sulfonate, toluene sulfonate, cumene sulfonate, an alcohol, a short chain fatty acid, in low level concentration, aids in improving water dispersibility of the N-C₇ to C₁₀ alkyl pyrrolidones since they are readily solubilized in aqueous solutions of pH 6 or less.

The hydrophilic and oleophilic properties of certain members in the present group of pyrrolidones makes them selectively suitable for certain applications. Specifically, the oleophilic members are suitable for extraction processes, e.g. in the recovery of residual material or in the fractional separation of a compound from aqueous solution such as the separation of a drug under conditions where a low temperature must be employed to prevent its decomposition. In such cases, the present lactams complex with the drug and the resulting drug-complex is easily separated by heating the complex above its cloud point to precipitate the drug which is then skimmed off the surface as the lactam layer.

The members which exhibit only partial water solubility, such as the C₁₀ and C₁₄ alkyl species, are nevertheless important as non-toxic time release surfactant-complexants for drugs, insecticides and the like which obviate the need for extraneous adducts or other surfactants in the formulation.

While generally the present N-alkyl pyrrolidones are relatively low foamers and while the N-tetradecyl species is particularly useful in stabilizing foaming of a commercial detergent compositions at temperatures in the range of 40-50°C., as in household and industrial cleaning solutions, it is surprising to find that the N-octyl and

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the N-decyl pyrrolidones augments and significantly increases foaming and foam stabilization of detergent solutions, such as those employed in a laundry or dishwashing solution, bar soap, cleansing cream, hair mousse etc. Generally for domestic use, high foaming dishwashing fluids are preferred; whereas for industrial use low foamers are in demand. In laundry detergents lower sudsing formulations are desired for better rinsing. By judicious selection of the proper member of surfactants herein disclosed, both demands can be satisfied. These lower alkyl species are also useful in flotation deinking or other flotation processes where high foam levels are desired, in forming as foam markers and in plastic coating and foaming applications, e.g. for enhancement of polyurethane foam. The excellent wetting properties of the present lactams also recommends their use in cosmetic creams, emollients, soaps and shampoos.

In addition, the present compounds impart anti-static and softening properties to fabrics during a laundry drying or washing cycle. It is discovered that residual anti-static protection is provided even when the present surfactants are added only in the washing cycle. Accordingly these pyrrolidones can be added to a detergent formulation or can be applied directly to a fabric to coat fibers or to a cellulosic material e.g. as a anti-static spray, for rugs or clothing or can be used as paper coated strips which are added to a dryer. Soil release and anti-soil deposition on fabrics is also imparted when the fabrics are coated, washed or otherwise contacted with the pyrrolidones of this invention. Instant products also can be applied to printing rollers or any equipment where slippage, friction or other factors build-up a considerable electrostatic charge as for example in electronic equipment or equipment used for the manufacture of certain plastics. Additionally, the excellent thermal stability of the present lactams makes them useful as high

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temperature anti-static agents and permits their formulation into molten plastics such as polyethylene, polypropylene, nylon, mylar, etc. before extrusion without any degradation of the plastic. In such oleophilic matrices, the hydrophilic pyrrolidone migrates to the plastic surface and acts as an antistat and anti-blocking agent.

The complexing properties of the present pyrrolidones makes them particularly effective as biocidal washing agents employed for hospital, household or industrial use. A particular application would involve the complex formed between iodine and instant pyrrolidones as an effective antiseptic solution which can be used as such, e.g. as a spray, or can be added to a detergent solution to provide or enhance germicidal properties. Also, such complexes of the higher molecular pyrrolidones of C₁₄-C₂₀ alkyl species, having higher viscosity can be applied to wounds as a cream or as a flexible antiseptic bandage or can be formulated into other compositions to enhance viscosity.

Because of their excellent wetting, fixative and complexing properties the C₇ to C₂₀ alkyl-2-pyrrolidones are beneficially incorporated in formulations for perfume, or for insecticides, herbicides, plant growth regulants, pesticides, annelidicides, etc. as a fixing agent to retain the active agent on the surface of the plant membrane or close to the skin surface where applied. Also biocidal solutions using complexes of the present pyrrolidones with various drugs can be used in veterinary medications or as disinfectant washing solutions, such as a wash for range cattle to kill incipient bacteria while providing residual inhibition of future infections from ticks and other pests. The high skin and hair substantivity and moisturizing capability of

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the present surfactants particularly the C_{12} - C_{20} alkyl species makes them beneficial adducts in the cosmetic art for use in lotions, creams, shampoos and hair sprays. They also provide excellent hair hold and curl retention when employed in hair setting lotions.

The excellent wetting properties of instant lactams recommends them as outstanding surfactants for use in concentrate formulations of wettable powders, particularly fungicidal and herbicidal powders, which are subsequently diluted with water to form aqueous suspensions suitably employed as sprays for application on crops. These suspensions which include the lactam in the active concentrate formulation are distinguished by significantly improved stability over extended periods. The method for preparing the concentrate involves blending the active component, preferably having a particle size less than 25 micrometers, with an inert carrier, a dispersing agent to prevent flocculation and the lactam wetting agent to facilitate the suspension of particles. The blend is generally milled, eg. on a fly cutter mill operated at about 20,000 rpm, at ambient temperature for a period of from about 0.5 to about 5 minutes. This operation provides a wettable powder concentrate having a suspensibility greater than 75% and a wetting time of less than 3 seconds. Suitable fillers for the concentrate include clays, talc, silica powder, bentonite, and diatomaceous earth. The blends of anionic dispersing agent and the nonionic lactam with the wettable powder and filler ensures good storage stability and improves suspension properties upon dilution in the field. The present lactams are highly compatible with anionic dispersants commonly employed, such as Igepon T-77 (a sodium salt of fatty acid amide sulfonate), Blancol (the sodium salt of sulfonated naphthalene-formaldehyde condensate) and Marasperse N (a lignosulfonate). The

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lactams also guarantee good formulation and tank mixing capability with the most common active ingredients.

5 Certain synthetic fibers, for example polyesters, nylons, orlon and fiber blends, are difficult to dye because they provide insufficient ionic sites on which the dye can attack. Accordingly, dying of such fibers or fabrics is usually effected with disperse dyes which require no complexing function for color development. However, one objection to this type of dying is that the product does not possess high color fastness. The present nonionic surfactant/complexing lactams which lower surface tension and interact with both anionic and cationic substances, promotes acceptance of non-disperse dyes such as for example the acid dyes, cyanine dyes, anthraquinone dyes, guinoline dyes and thiazole dyes on relatively difficult dyeable materials while simultaneously providing faster release and higher exhaust of the dye from carrier and onto the textile substrate.

10 Additionally, the alkyl pyrrolidones are beneficially combined with polyvinylpyrrolidone complexes and other drug carrying complexes, to transfer the drug from the complex to the skin and to promote skin penetration for more effective use of the drug. In a similar manner the present lactams provide good dye penetration in fabrics, particularly for penetration of non disperse dyes in polyester fabric.

20 The linear C₇ to C₂₀ alkyl groups bonded to the heterocyclic nitrogen provide compounds which, in addition to their surfactant and complexing capability, are also excellent emulsifiers e.g. for paraffinic oil used in the metalworking industry.

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The present lactams are also useful in the preparation of emulsifiable concentrates of agricultural chemicals which, when added to water, form a sprayable oil-in-water emulsion having dispersed phase droplets in the range of from about 0.1 to about 5 micrometers. Such an emulsion provides a uniform and accurate application of the active ingredient, eg. on the crops, and ensures uniform spreading and wetting under normal spray and weather conditions to form such emulsifiable concentrates. The lactams of this invention are combined with at least one other amphoteric, anionic, non-ionic or cationic surfactant in a weight ratio of between about 1:10 and about 1:0.8 preferably between about 1:9 and about 1:1 at 15-30°C. Suitable co-surfactants include EMULPHOR® EL-620 (polyethoxylated av. 30 castor oil); EMULPHOR® EL-719 (polyethoxylated av. 40 castor oil); IGEPAL® CO-630 (ethoxylated av. 9 nonylphenol); IGEPAL® CO-530 (ethoxylated av. 6 nonylphenol); KATANOL® L-2 (trichlorobenzene), MIRANOL® DN (a stearoamphoacetate); SPAN® 40 (sorbitan monopalmitate), ANTARON® (a carboxyl cocoimidazoline), FENOPON® (coconut or myristic acid ester of sodium isethionate) and alkylamine guanidine polyoxyethanol.

Such emulsifiable concentrates are particularly useful in the preparation of herbicidal, fungicidal and insecticidal stable or fast breaking emulsions.

The anti-corrosion properties of instant pyrrolidones make them valuable corrosion inhibitors in oil well drilling acidification and many other fields such as prevention of corrosion of dairy equipment as well as in petroleum processing and acidizing and in plating baths. Aerosol sprays combining the surfactant, detergency and anti-corrosive properties of the present lactams, particularly the pyrrolidone lactams, can be used for cleaning and protection of automobiles, trucks, etc.

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The C₈-C₁₂ alkyl species are particularly effective against corrosion by inorganic acids, e.g. HCl, phosphoric acid, sulfuric acid by reason of their complexability both with the acid and with the metal oxides formed by corrosion. The N-alkyl-2-pyrrolidones herein described are absorbed on the surface of a metal and provide a hydrophobic layer between the metal and the attacking acid, thus protecting the metal from acid corrosion.

The pyrrolidone products of this invention are also excellent viscosity builders, the higher molecular weight species being outstanding. Accordingly, these pyrrolidones can be added to liquid formulations to provide gels or pastes. This application is extremely desirable where noxious or skin irritating liquid chemicals of low viscosity are employed. For example, gels or pastes of strong acids can be provided to eliminate splattering or fuming and more viscous compositions containing the active chemical can be applied for retention on vertical surfaces, e.g. to effect rust removal.

The electron rich N-alkylphenyl-pyrrolidones are used to coat electron deficient surfaces, such as polyester films and fibers. These aryl pyrrolidones provide excellent adhesion and reduced amounts can be used to obtain a satisfactory microthin coating.

Because of their surfactant properties, the present pyrrolidones also hinder the formation of hard water precipitates. Still further these products, which possess high plastic substantivity, provide internal and external lubricity to polymeric products such as those which are made from or contain polystyrene, polyethylene, polypropylene, polyvinyl chloride, nylon, cellulose acetate, polyvinyl acetate, phenolic resins, polyvinyl pyrrolidone, etc. Surface lubricating effects on metals

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have also been noted. Selection of individual N-alkyl pyrrolidones of the C₇-C₂₀ alkyl species or combinations thereof provide the effects mentioned above in finished formulations and other effects which will become apparent from the present disclosure.

Still another field in which the present lactams find application is in dry cleaning. Dry cleaning solvents generally fall into two categories, namely the petroleum solvents and the halogenated solvents which include Stoddard solvent (a petroleum distillate between gasoline and kerosene), carbon tetrachloride, trichloroethylene, perchloroethylene, fluorinated hydrocarbons, 104F solvent, etc. Although these solvents are satisfactory for the removal of fatty type soils, many water soluble spots and stains, eg. tea, fruit, wine, ink and beer stains, are not removed. However, when the present solvent soluble lactams are added to the formulation such water insoluble stains are easily removed. These lactams, particularly the pyrrolidones herein defined, complex with acidic molecules, labile protons, polarizable molecules and color forming components. They also complex with odor causing components in human perspiration this minimizing or eliminating odor retained in clothing including polyester fabrics. The present lactams are also efficacious in removing soil and stains when added to a standard laundry detergent. The effective amount of lactam incorporated in dry cleaning or laundry detergents for the above purposes is generally at least 1% by weight, preferably between about 2% and 50% by weight of the total formulation. As a specific spot and stain remover, however, the present lactams, particularly the pyrrolidones can be used individually or in admixture in 100% concentration with no additive. For effective stain removal, usually an amount which wets the entire stain will suffice to give desired results.

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In view of the diverse fields of application in which the present compounds are beneficially employed, it will be appreciated that widely varying amounts of these compounds can be used to fulfill their requirements and other functions which may become obvious. Generally, between 100% and about 0.001% of these surfactants depending upon their use as a compound per se or as an additive to an existing formulation will become apparent. More specifically, when one or more of the present products is used as an anti-static spray or stain remover, up to 100% of the active ingredient can be the present pyrrolidone or a blend thereof. On the other hand, when the present product is an additive in a laundry, dishwashing or cosmetic formulation, its concentration can be as low as 0.001%. For complexing, the concentration of pyrrolidone depends entirely on the chemical nature of the compound and the amount of drug, agricultural chemical or other active component one desires to incorporate. Generally, the amount employed is within the mole ratio range of between about 0.5:1 and about 99:1 lactam to active component and an amount at least sufficient to retain the beneficial characteristics of the lactam but not more than the amount needed to preserve the effect of the active component being complexed.

Shampoo and hair care applications generally use from about 1% to about 20% by weight of the present lactams based on total compositions; whereas skin care and other cosmetics may employ as little as 0.001 wt. %, preferably between about 2% and about 0.01% by weight of the lactam based on total composition. As viscosity builders, up to 80% of the product may be present in the formulation. Most often, the amount of lactam product employed is the same or somewhat less than that amount used for agents having a similar property in the same field of application.

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Having thus described the invention, reference is now had to the following examples which set forth preferred embodiments but which are not to be construed as limiting to the scope of the invention as more broadly described above and as set forth in the appended claims. It should be recognized that these examples are presented only to more specifically describe the invention, to exemplify preferred embodiments and to provide representative examples of use from which other uses and applications will become apparent.

EXAMPLE I

Into a stainless steel autoclave was introduced n-octylamine (2342 g) and butyrolactone (1704 g) in a mole ratio of about 1:1.1. The autoclave was sealed and 100 psig of nitrogen applied and the contents heated to 275°C. and held for 8 hours during which time the reaction mixture was agitated and the pressure increased from atmospheric to about 480 psig. The reaction produced a liquid product which was recovered from the autoclave and distilled to produce 98.5% pure clear water white liquid N-octyl-2-pyrrolidone in a yield of 98% and having a boiling point of 118°C. at 0.5 mm of Hg, a viscosity of 8 cps, and a pH of 6.5 (10% in 50/50 isopropyl alcohol-water).

EXAMPLE II

The procedure described in Example I was repeated except that decylamine was substituted for n-octylamine and the reaction product was distilled to provide 99.3% pure clear, water white liquid N-decyl-2-pyrrolidone in a yield of 98% and having a boiling point of 120°C. at 0.2 mm of Hg, a viscosity of 12 cps, and a pH (10% in 50/50 isopropyl alcohol-water) of 5.4.

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EXAMPLE III

The procedure described in Example I was repeated except that dodecylamine (Armeen 12D) was substituted for n-octylamine and the reaction product was distilled to provide 99.2% pure clear, water white liquid N-dodecyl-2-pyrrolidone in a yield of 99% and having a boiling point of 145°C. at 0.2 mm of Hg, a viscosity of 17 cps, a cloud point (10% H₂O soln.) of 15-19°C., and a pH (10% in 50/50 isopropyl alcohol-water) of 7.3.

EXAMPLE IV

The procedure described in Example I was repeated except that tetradecylamine was substituted for n-octylamine and the reaction product was distilled to provide 96.9% pure clear, water white liquid N-tetradecyl-2-pyrrolidone in a yield of 95% and having a boiling point of 190°C. at 1.0 mm of Hg, a viscosity of 2 cps, a cloud point (10% H₂O soln.) of 33°C., and a pH (10% in 50/50 isopropyl alcohol-water) of 5.9.

EXAMPLE V

The procedure described in Example I was repeated except that hexadecylamine (Armeen 16D) was substituted for n-octylamine and the reaction product was distilled using a hot water condenser and was discharged at about 70°C. to provide 94.5% pure N-hexadecyl-2-pyrrolidone solid in a yield of 90% and having a boiling point of 180°C. at 0.1 mm Hg.

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EXAMPLE VI

5 The procedure described in Example I was repeated except that octadecylamine (Armeen 18D) was substituted for n-octylamine and the product was stripped of water and excess butyrolactone and was discharged at about 75°C. to provide 96% pure N-octadecyl-2-pyrrolidone solid in a yield of 96%.

EXAMPLE VII

10 The procedure described in Example I is repeated except that eicosylamine is substituted for octylamine and the product distilled using a warm water condenser and was discharged at about 85°C. to provide 95% pure N-eicosyl-2-pyrrolidone solid in a yield of about 90%.

EXAMPLE VIII

15 The procedure of Example I was repeated except that 2-ethylhexylamine was substituted for n-octylamine. The reaction product was distilled to provide 99.5% of pure clear water white liquid N-2-ethylhexylpyrrolidone-2, boiling at 92-97°C. at 0.06 mm Hg in 96% yield.

EXAMPLE IX

20 The procedure of Example I was repeated except that Primene 81R (a tertiary C₁₂-C₁₄ alkyl primary amine) was substituted for n-octylamine. The reaction product was distilled to provide 97% pure liquid
25 alkylpyrrolidone product in 40% yield. This product boils at 112-115°C. at 0.7 mm Hg.

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EXAMPLE X

5 The procedure of Example I was repeated except that t-octyl primary amine was substituted for n-octylamine. The reaction product was distilled at 98-102°C. and 0.5 mm Hg to provide 99.3% pure product in 50% yield.

EXAMPLE XI

10 The procedure of Example I was repeated except that α -naphthyl amine is substituted for n-octylamine. The reaction in this case yields a solid product which is recovered from the autoclave and distilled to provide 99% pure product in 50% yield. Product has a melting point of 110-112°C.

15 The same procedure is employed to produce N-alkyl naphthyl lactams by substituting the appropriate N-alkyl naphthyl amine for naphthyl amine in this example.

EXAMPLE XII

20 The procedure of Example I was repeated except that aniline is substituted for n-octylamine. The product is recovered from the autoclave and distilled at 123°C. under 0.2 mm Hg to provide 99% pure product in 50% yield.

25 The same procedure is employed to produce N-alkylaniline lactams by substituting the appropriate N-alkylaniline for aniline in this example.

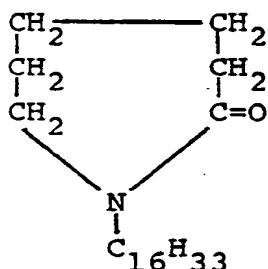
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EXAMPLE XIII

The procedure of Example I was repeated except that cocoamine distillate was substituted for n-octylamine. The reaction product was stripped of water and excess butyrolactone and discharged. The product was obtained in 96% purity and 97% yield.

EXAMPLE XIV

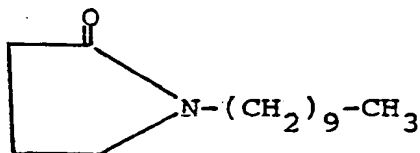
The procedure of Example I is repeated except that caprolactone is substituted for butyrolactone and hexadecyl amine is substituted for n-octylamine. The product,



is recovered in 90% yield and purity.

EXAMPLE XV

Preparation of N-n-decyl-2-pyrrolidone

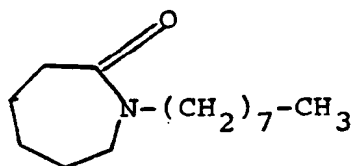


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5 In a glass reactor, 19 g (0.22 M) of γ -butyrolactone and 34.6 g (0.22 M) of n-decylamine are mixed and heated to 180° in a round bottom flask equipped with a condenser and a Dean-Stark trap for 22 hours. The dark brown reaction mixture is distilled at reduced pressure to yield 50 g (82.5%) of colorless product; b.p. 150°-155°/0.5-1 mm Hg.

EXAMPLE XVI

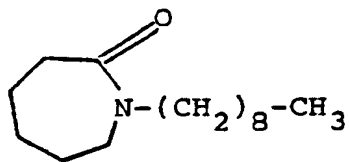
10 Preparation of N-n-octyl-2-caprolactam having the formula



15 Following Example XV, heating 17.5 g (0.153 M) of 6-hexanolactone and 22 g (0.17 M) of 1-aminooctane at 180° for 29 hours gives 9 g (27%) of product; b.p. 155°-160°/0.5 mm Hg.

EXAMPLE XVII

Preparation of N-n-nonylcaprolactam having the formula

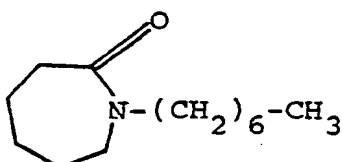


20 Following Example XV, heating 23 g (0.2 M) of 6-hexanolactone and 28.65 g (0.2 M) of 1-aminononane at 180° for 20 hours gives 11.5 g (26%) of product; b.p. 155°-165°/0.6 mm Hg.

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EXAMPLE XVIII

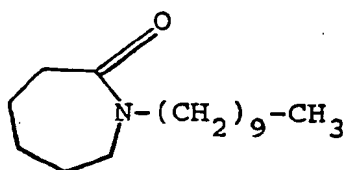
Preparation of N-n-heptyl-2-caprolactam having the formula



5 In a 1 liter 3-neck flask equipped with a water condenser, an addition funnel and a mechanical stirrer is placed 10.2 g of 50% sodium hydride-mineral oil dispersion (5.1 g NaH, 0.2125 M) and 150 ml of petroleum ether. The suspension is momentarily stirred and then sodium hydride is allowed to settle. Most of the petroleum ether is pipetted out and 200 ml of dry toluene is added, after which a solution of 20 g (0.177 M) of 2-caprolactam-2-one in 100 ml of dry toluene is added. The mixture is refluxed for 1 hour and then cooled to room temperature. 15 A solution of 38.8 g (0.25 M) of 1-bromoheptane in 100 ml of dry toluene is added dropwise under stirring. Upon completion of the addition, the mixture is warmed to 80°-100° and the temperature was maintained for 4 hours. The reaction mixture is then heated to reflux for 18 hours to give 90% of a colorless product having a b.p. of 20 155°-158°C. at 0.5 mm Hg.

EXAMPLE XIX

Preparation of N-n-decylcaprolactam having the formula

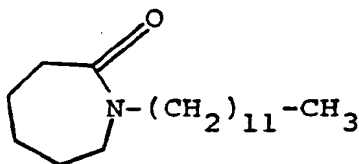


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Following Example XVIII, 10.2 g of 50% sodium hydride-mineral oil dispersion (5.1 g NaH, 0.2125 M), 20 g (0.177 M) of azacycloheptan-2-one and 44.2 g (0.2 M) of 1-bromodecane on 19 hr. reflux gives 38 g (84.7%) of product; b.p. 158°-163°/0.25-0.3 mm Hg.

EXAMPLE XX

Preparation of N-n-dodecylcaprolactam having the formula



Following Example XVIII, 15.3 g of 50% sodium hydride-mineral oil dispersion (7.65 g NaH, 0.319 M), 30 g (0.266 M) of azacycloheptan-2-one and 66.1 g (0.265 M) of 1-bromododecane on 20 hours reflux gives 58.9 g (80%) of colorless product; b.p. 175°-180°/0.3 mm Hg.

EXAMPLE XXI

SURFACE TENSION

Surface tension measurements in triplicate were made for each of the N-alkylpyrrolidone species listed below in Table 1, using a Fisher Surface Tensiomat (Model #21, Der Nouy Ring Tensionometer). Each experiment was carried out as follows.

Distilled water solutions at concentrations noted in Table 1 were prepared for each of the following surfactants in 100 ml glass flasks. The solutions were stirred for about 15 minutes until homogeneous solutions were obtained. The surface tensions of these solutions were then measured.

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The averaged results of the above tests are reported in Table 1.

TABLE 1

Surface Tension of Aqueous Solutions at 25°C. (dynes/cm)

5	Product	<u>% Solution in Distilled Water</u>				
		<u>1.0%</u>	<u>0.1%</u>	<u>0.01%</u>	<u>0.001%</u>	<u>0.0001%</u>
	Octyl pyrrolidone	--	30.4	54.25	68.4	--
	Decyl pyrrolidone	27.6	27.5	27.8	51.0	--
	Dodecyl pyrrolidone	--	26.7	27.6	29.9	55.4
10	Tetradecyl pyrrolidone	--	26.4	26.5	30.4	47.5

Surface tension data indicates that these products have strong surface activity, i.e., lowering the surface tension of water.

From the surface tension data, the surface excess concentration Γ (moles/cm²), area/molecule (a_m Å²)* at the interface and efficiency of adsorption, (pC_{20})** at the solution/air interface were computed by use of appropriate Gibb's Adsorption Equation:

$$20 \quad \Gamma = \frac{-d\gamma}{d \log c_T} / 2.303 RT$$

where Γ = surface excess concentration (moles/cm²)
 $d\gamma$ = change in surface or interfacial tension of the solvent

$$R = 8.31 \times 10 \text{ ergs mol}^{-1}$$

25 c = Molar concentration of solution

T = Absolute Temperature

* Absorption Å²

** The negative logarithm of the surfactant concentration required to lower surface tension by 20 dynes/cm.

EXAMPLE XXII

5 The critical micelle concentration (CMC), the surface concentration at the liquid-air interface (Γ), the area of the test molecule at the interface (a_m) and the effectiveness of adsorption at the interface ($pC-20$) were determined on the above surface tension data reported in Example XXI, Table 1. These properties are reported in Table 2 below.

TABLE 2

SURFACE ACTIVITY PARAMETERS CALCULATED
FROM SURFACE TENSION MEASUREMENTS

<u>Molecule</u>	<u>CMC (ml⁻¹)</u>	<u>$\Gamma_m(\text{mcm}^{-2} \times 10^{10})$</u>	<u>$a_m (\text{\AA})^2$</u>	<u>pC-20</u>
N-n-octyl pyrrolidone	-	4.2	39.5	3.20
N-n-decyl pyrrolidone	4.4×10^{-4}	4.6	36.1	4.51
N-n-dodecyl pyrrolidone	4.7×10^{-5}	4.5	36.9	5.27
N-tetradecyl pyrrolidone	5.7×10^{-5}	3.1	53.6	6.25
lauryl dimethyl amine oxide*	2.1×10^{-3} *	2.8	59.3	5.23

* Literature value $\Gamma_m = 3.5 \times 10^{-10} \text{ m cm}^{-1}$, $a_m = 47 \text{\AA}^2$ (SURFACTANTS AND INTERFACIAL PHENOMENA by Rosen, page 68).

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More specifically, the CMC of the N-(n alkyl) pyrrolidones tested show a minimum (4.7×10^{-5}) at the C_{12} chain length. Using the empirical equation for n-lauryl (C_{12}) alcohol ethoxylate,

$$\log C_{CMC} = A^1 + B^1 n$$

where $A^1 = 4.4$, $B^1 = 0.046$, and n = number of ethylene oxide at 23°C ., the pyrrolidone ring would be expected to behave as 2 ethylene oxide units. However, the pyrrolidone ring is actually producing results equivalent to about 6 ethylene oxide units, i.e. at 25°C ., $n\text{-}C_{12}H_{25}$ (ethylene oxide)₄ OH (CMC = 4×10^{-5}) and $n\text{-}C_{12}H_{25}$ (ethylene oxide)₇ OH (CMC = 5×10^{-5}). The hydrophilic-lipophilic balance (HLB) of N-n-dodecyl pyrrolidone, based on the assumption that the pyrrolidone ring simulates 2-6 ethylene oxide units is 6.5 - 11 using the equation $\% \text{ ethylene oxide} / 5 = \text{HLB}$. The low apparent HLB suggests the material would be a good water-in-oil emulsifier, e.g. in skin care products.

The surface concentration Γ , in moles per cm^2 indicates the maximum surface concentration of surfactant, i.e. at equilibrium above the CMC, and is obtained by an estimation of a constant $d\gamma/dc^*$ from the surface tension vs. concentration. The N-(n alkyl) pyrrolidones obtain a maximum T_m ($4.6 \times 10^{-10} \text{ m cm}^{-2}$) at $N\text{-}C_{10}H_{21}$ chain length. In general, the T_m for all of the N-n alkyl pyrrolidones is very high in comparison to all, but the less soluble alcohol ethoxylates, i.e. $N\text{-}C_{12}H_{25}$ (ethylene oxide)₄ OH ($T_m = 3.8 \times 10^{-10} \text{ m cm}^{-2}$) $n\text{-}C_{16}H_{33}$ (ethylene oxide)₆ OH ($T_m = 4.4 \times 10^{-10} \text{ m cm}^{-2}$). The mono disperse nature of the hydrophobe and hydrophile is believed to contribute to high surface concentrations by maximizing entropy of packing.

* $d\gamma/dc$ = the change in surface tension with respect to the change in surfactant concentration.

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As the interface becomes saturated with surfactant molecules, one would expect that within a homologous series, the longer hydrophobic chains would sterically hinder more surfactant molecules from positioning themselves at the interface. The decrease in T_m of the C_8H_{17} vs. the $C_{10}H_{23}$ chain is attributed to the hydrophile/hydrophile interactions of the n-octyl-2-pyrrolidone. Above the $C_{10}H_{23}$ alkyl chain length, normal steric hinderance of the hydrophobes determines T_m .

The area per molecule (\AA^2) is relative to the T_m , in a comparison of a homologous series. Generally, the area per molecule increases with increasing chain length of the N-(n alkyl) pyrrolidones; however, the larger am of the n-octyl pyrrolidone is attributed to an attraction of pyrrolidone moieties not balanced by the alkyl chain attractions. The low am of the N-(n alkyl) pyrrolidones approaches the theoretical size (20\AA^2) for an aliphatic chain oriented perpendicular to the interface.

The negative logarithm of the surfactant concentration required to lower the surface tension by 20 dynes/cm⁻¹ can be used as a measure of the efficiency of adsorption since Γ is approaching the maximum value. The N-(n alkyl) pyrrolidones actually show a progressively higher efficiency of adsorption as the alkyl chain is increased.

EXAMPLE XXIII

FOAM TESTING

(A) RELATIVE FOAMING PROPERTIES - ROSS MILES FOAM TEST

To determine the sudsing characteristics of the pure compounds, Ross Miles sudsing tests were conducted on 1.0% surfactant solutions in distilled water at ambient

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(~25°C.) temperature. The results indicate that the N-alkylpyrrolidones are low foamers, when compared to fatty alkyl benzene sulfonates, fatty alkyl ethylene oxide sulfates, fatty alkyl dimethyl amine oxides, and fatty alkyl alkanol amides. The low sudsing is maximized at the C₁₂ alkyl chain length.

Solutions of 1% by weight of for each surfactant in distilled water noted below were made up. Into a graduated glass cylinder was poured 50 ml of a given 1% solution and another 200 ml of the same solution was dropped into the first 50 mls. from a height of 90 cm. The foam height developed in the cylinder was measured by the cylinder graduations. The results of these tests are reported in following Table 3.

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TABLE 3

Ross Miles Sudsing Data in ml of foam (1.0% Surfactant in Distilled Water at Ambient Temperature, 25°C. - Average 2 Runs)

	<u>Product</u>	<u>Breakdown Time</u> (Min.)			<u>Total Breakdown</u> (Min.)
		<u>0</u>	<u>5</u>	<u>10</u>	<u>Initial-Final</u>
0	Octyl Pyrrolidone	25.0	6.0	5.5	19.5
	Decyl Pyrrolidone	24.5	24.5	24.0	0.0
	Dodecyl Pyrrolidone	26.5	26.5	26.5	0.0
	Tetradecyl Pyrrolidone	18.0	18.0	18.0	0.0
5	Dodecyl Benzene Sulfonate	175	166	146	28.0
	Alipal CO-436(1)	167	166	166	2.5
	Gafamide CDD-518(2)	160	160	160	0.0
	Ammonyx MO(3)	167	167	167	0.0
20	Ammonyx LO(4)	188	188	185	3.5
	Aromox DMC-W(5)	191	191	190	2.0
	Control (Dist. (Water))	0.0	0.0	0.0	0.0

(1) The ammonium salt of sulfated nonylphenoxypoly (ethyleneoxy) ethanol (supplied by GAF Corp.).

25 (2) Coconut oil diethanolamine condensate (supplied by GAF Corp.).

(3) Myristyl dimethylamine oxide.

(4) Lauryl dimethylamine oxide.

(5) Dimethyl cocamine oxide.

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(B) SUDS BOOSTING PROPERTIES

Since the N-(n alkyl)-2-pyrrolidones are neutral molecules at pH 7 and above, they should increase the density of the anionic surface layer and act as suds boosters for both linear alkyl benzene sulfonates and ethylene oxide based products. Ross Miles sudsing data (0.5% surfactant/0.5% suds booster) indicate the N-(n alkyl) pyrrolidones are highly effective in boosting and stabilizing the foam of linear alkyl benzene sulfonates. The N-decylpyrrolidone appears to be the most efficacious of the compounds tested in boosting the foam of Alipal CO-436. This property of the present N-(n alkyl) pyrrolidones is important in laundry, dishwashing, soap bars, cleansing creams, flotation deinking, and use as foam markers, etc. The sudsing data is shown in Table 4.

TABLE 4
ROSS MILES SUDS TESTS- BOOSTING/STABILIZING OF LINEAR DODECYL BENZENE
SULFONATE (LAS) AND NONYLPHENOL ETHYLENE OXIDE-S* BY N-n-ALKYL PYRROLIDONES
(0.5% surfactant/0.5% pyrrolidone booster at ambient temperature)

Surfactant	Suds Booster	Foam Height (mm)			Total Breakdown (Min) from initial to final
		Breakdown (min.)			
		0	5	10	
LAS	N-C ₈ alkyl-2-pyrrolidone	195	194	184	10.5
LAS	N-C ₁₀ alkyl-2-pyrrolidone	204	202	202	2.0
LAS	N-C ₁₂ alkyl-2-pyrrolidone	163	163	163	0.0
LAS	N-C ₁₄ alkyl-2-pyrrolidone	127	127	127	0.0
LAS	none	175	166	147	28.0
ALIPAL CO-436	N-C ₈ alkyl-2-pyrrolidone	185	183	183	2.0
ALIPAL CO-436	N-C ₁₀ alkyl-2-pyrrolidone	202	200	199	2.5
ALIPAL CO-436	N-C ₁₂ alkyl-2-pyrrolidone	176	176	176	0.0
ALIPAL CO-436	N-C ₁₄ alkyl-2-pyrrolidone	148	148	148	0.0
ALIPAL CO-436	none	167	166	166	1.5

* ethylene oxide sulfate salt

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(C) FOAM "BOOSTING" IN MANUAL DISHWASHING FORMULATION

To determine how the present N-(n-alkyl)-2-pyrrolidones would function in a consumer-type dishwashing product, they were tested at 40°C. in the basic formula shown in Table 5.

TABLE 5Model Dishwash Formula

	<u>Component</u>	<u>Wt. %</u>
	Water	65.0
10	Ethanol	11.7
	* Alfonic 1412-A (100% act.)	13.3
	** Alfonic 1412-40	1.1
	NaCl	0.9
	N-alkyl-pyrrolidone	
15	suds "booster"	7.5
	Misc. (from Alfonic 1412-A)	<u>0.5</u>
	TOTAL	100.0

The data indicates that without soil at ambient temperature ($\sim 25^{\circ}\text{C}.$), in 100 ppm hardness ($3/2$, Ca/Mg), the suds booster efficiency of the N-(n-alkyl) pyrrolidones (from N- C_8 to C_{14} alkyl) decreased with increasing chain length. The octyl pyrrolidone showed higher suds levels, increased from 147 mm to 158 mm. The C_{12} and C_{14} pyrrolidones decreased the suds level of the model formula from 147 to 138 and 147 to 118 mm respectively.

* a sulfated, ethoxylated alcohol derivative - an anionic biodegradable surfactant

** 40% ethoxylated linear alcohol - a nonionic biodegradable surfactant

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(D) EFFICIENCY OF FOAM BOOSTING IN PRESENCE OF SOIL

A soil mixture of 47 wt. % bleached flour, 48 wt. % of partially hydrogenated vegetable oil shortening (Crisco) and 5 wt. % of oleic acid was mixed and heated to 50°C. to form a slurry. To an aqueous solution containing 0.15% N-alkyl-2-pyrrolidone in 100 ppm hard water was added 0.1% of the above slurry and mixed for 10 minutes at 40°C. The results of this series of tests is reported in Table 6.

TABLE 6

Ross Miles Sudsing Data at Ambient Temperature
(0.15% Product Use Level - Table 5 composition
100 ppm water hardness, 3/2-Ca/Mg)

	<u>Suds Booster</u> <u>(7.5% of Product)</u>	<u>Foam Height (mm)</u> <u>Breakdown Time</u> <u>(Min.)</u>		
		<u>0</u>	<u>5</u>	<u>10</u>
	n-octyl pyrrolidone	158	158	158
	n-decyl pyrrolidone	154	153	153
	n-dodecyl pyrrolidone	138	138	138
	n-tetradecyl pyrrolidone	118	118	118
	Product with no booster	147	147	147
	Commercial "Joy"	154	154	154

Of the samples tested, after 5 minutes no foam breakdown had occurred.

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EXAMPLE XXIVWARING BLENDER LATHER TEST

Several samples to be tested were prepared in 1500 ml glass beakers by mixing 47.95 wt. % of deionized water at 60°C. with 50 wt. % surfactant and 0.6 wt. % of the foam booster (100% active) to be tested while agitating constantly with an electric stirrer. The resulting solution was allowed to cool to 40°C. whereupon 0.05 wt. % of Kathon CG* preservative was added. Stirring was continued until the solution cooled to room temperature.

A control solution was prepared for each surfactant used in the same manner except that foam booster was omitted from the control formulations and 49.95 wt. % of deionized water was employed.

Each of the above solutions was subjected to a Waring Blender Lather Test which entailed introducing 100 ml of the test or control solution at 25°C. into a high speed Waring Blender operated at 21,000 RPM. Following agitation for one minute, the foam is transferred to a 1000 ml. graduated cylinder and the foam volume in ccs recorded. The results of these tests are reported in the following Table 7.

Although the initial foam height of the control is greater the foam comprises large loose bubbles, hence the foam density is significantly lower. Also the foam of the control collapses within about one minute; whereas the foams obtained with the present lactams comprise small densely packed bubbles and are stable beyond 5 minutes.

* N-methylchloroisothiazolinone + methylisothiazoline

TABLE 7

Surfactant= lauryl sulfate	
FOAM BOOSTER	FOAM VOLUME (cc/100 ml test solution) Initial
N-dodecyl-2-pyrrolidone	460
coconut diethanol amide (GAFAMIDE CDD-518)	425
lauryl dimethylamine oxide (Ammonyx LO)	450
cocamidopropyl betaine	485
cocamidopropyl hydroxy sultaine	460
Control	600
Surfactant= Na laureth sulfate (3moles ethylene oxide)	
FOAM BOOSTER	
N-dodecyl-2-pyrrolidone	435
coconut diethanol amide	420
lauryl dimethylamine oxide	430
cocamidopropyl betaine	515
cocamidopropyl hydroxy sultaine	415
Control	500
Surfactant= ammonium lauryl sulfate	
FOAM BOOSTER	
N-dodecyl-2-pyrrolidone	215
coconut diethanol amide	140
lauryl dimethylamine oxide	170
cocamidopropyl betaine	325
cocamidopropyl hydroxy sultaine	410
Control	500

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EXAMPLE XXVWETTING TEST - DRAVE METHOD

Into 99.9 ml of distilled water, 0.1% by weight of the N-alkyl-2-pyrrolidone noted in Table 9 was dissolved. The resulting aqueous solution is poured into a one liter glass volumetric flask and 5 g of 100% cotton yarn weighted with a 3 g hook was dropped into the beaker. The time required for the yarn to sink below the surface of the aqueous wetting solution is reported in Table 8. Wetting tests for each of the following N-alkyl-2-pyrrolidone species were run in triplicate.

TABLE 8DRAVE'S WETTING DATA AT 25°C. \pm 1°C.

	<u>Product</u>	<u>Time in Seconds</u>
15	N-Octyl pyrrolidone	4.4
	N-Decyl pyrrolidone	68.5
	N-Dodecyl pyrrolidone	93.5
	N-Tetradecyl pyrrolidone	109.3

The wetting efficacy of the N-n C₇ to C₂₀ alkyl pyrrolidone series decreases with increasing molecular weight. Of the series, the C₈- and probably the C₇- alkyl compounds could be considered excellent wetting agents (4.4 seconds wetting at 0.1% for C₈-alkyl-2-pyrrolidone).

EXAMPLE XXVISOLUBILITY OF N-ALKYL PYRROLIDONES

Separate 10% by weight solutions of N-alkyl-2-pyrrolidone in the following liquids were made up in 50 ml glass beakers and the solubilities at about 25°C. were noted and reported in following Table 9.

TABLE 9

10% PRODUCT SOLUBILITY AT 25°C ± 1°C

SOLVENT	N-octyl-2- pyrrolidone	N-decyl-2- pyrrolidone	N-dodecyl-2- pyrrolidone	N-tetradecyl-2- pyrrolidone
Water	I	PS*	S*	S*
Acetone	S	S	S	S
Ethanol	S	S	S	S
Xylene	S	S	S	S
Heptane	S	S	S	S
Paraffin Oil	S	S	PS	S
Stoddard Solvent (1)	S**	S	PS	S
Perchloroethylene	S	S	S	S

I= insoluble; PS= partially soluble (cloudy translucent); S= soluble, clear

(1) US Bureau of Standards specification- a clear, water white petroleum distillate

*Soluble with slight haze

**Very slight precipitation

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The data indicates that alkyl pyrrolidones are soluble in various types of solvents. Although octyl pyrrolidone is insoluble at room temperature in water, decyl pyrrolidone is slightly soluble and dodecyl pyrrolidone and tetradecyl pyrrolidone are completely soluble in water at 10% concentration. However, dodecyl pyrrolidone and tetradecyl pyrrolidone exhibit iridescence phenomena at the 1% to 2% level in aqueous media (violet color at 2% and greenish at about 1%) which was destroyed in the presence of small amount of electrolyte.

It was also observed that 10% aqueous solution of tetradecyl pyrrolidone and dodecyl pyrrolidone become cloudy at about 33-34°C. and 31-32°C. respectively exhibiting cloud point phenomena.

The aqueous solubility of the dodecyl and tetradecyl products is interesting, i.e. below 1% (wt. %) solute, the solutions are clear. Between 1 and 2% the solutions exhibit a scattering pattern (breaks light into its visible spectrum) for visible light indicating a regular geometric pattern which could be regular crystalline spacing (lamellar type) or a monodisperse liquid crystal particle. Above 2% the solutions again become clear, suggesting a self dissolving effect (loss of regular geometry). This effect once again demonstrates the purity of these compounds. It is postulated that blends of n-alkyl chains would decrease the ability of these materials to form lamellar type structures and increase the aqueous solubility of the less soluble compounds (C_8 and C_{10}). Also it has been observed that low levels of electrolyte eliminate the "prism effects" shown by the 1-2% solutions of the C_{10} - C_{12} compounds.

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5 The solubility of the present pyrrolidones in both water and in nonpolar solvents such as heptane and perchloroethylene make them useful in nonaqueous applications such as dry cleaning detergents, solubilizers for water in fuels, and cleaners for the removal of solid fatty soil from hard surfaces.

EXAMPLE XXVII

10 The viscosities in cps of the following surfactants at various concentration in distilled water were measured at about 25°C. using a Brookfield Viscometer (LVT Model). The results of these tests are reported in following Table 10.

TABLE 10

VISCOSITY (CPS) AT VARIOUS WEIGHT % WATER CONCENTRATIONS

PRODUCT	100%	10%	8%	6%	4%	2%
N-octyl-2-pyrrolidone	8.0	-	-	-	-	-
N-decyl-2-pyrrolidone	12.0	-	-	-	-	-
N-dodecyl-2-pyrrolidone	17.0	20.0	16.0	12.5	7.5	6.5
N-tetradecyl-2-pyrrolidone	20.0	145.0	100.0	96.0	66.0	24.5
Ammonyx L0	-	7.0	-	-	-	4.0
Ammonyx M0	-	52.5	-	-	-	7.0
Control (distilled water)	1.0	-	-	-	-	-

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The above viscosity of alkyl pyrrolidones and their aqueous solutions were obtained using a #2 spindle at a speed of 30 rpm, and a #2 spindle at a speed of 60 rpm respectively.

5 Data indicates that viscosity of the (100% active) alkyl pyrrolidone increased as the carbon chain length of alkyl group increased.

Both tetradecyl and dodecyl pyrrolidone exhibit a significant thickening increase. The higher viscosities of the N-n alkyl pyrrolidones vs. dimethyl fatty amine oxides of similar chain length indicate that the micelles of the N-n alkyl pyrrolidones are lamellar or rod shaped as compared to spherical types for the amine oxides.

EXAMPLE XXVIII

VISCOSITY BUILDING

15 Several samples to be tested were prepared in 1500 ml glass beakers by mixing 47.9 wt. % of deionized water at 60°C. with 50 wt. % surfactant and 0.6 wt. % of the viscosity builder (100% active) to be tested while
20 agitating constantly with an electric stirrer. The resulting solution was allowed to cool to 40°C. whereupon 0.05 wt. % of Kathon CG preservative was added with stirring and the solution allowed to cool to room temperature.

25 A control solution was prepared for each surfactant used in the same manner except that viscosity builder was omitted from the control formulations and 49.95 wt. % of deionized water was employed.

30 The viscosity was measured in cps for each of the above solutions by adding 20% NaCl solution from a glass pipette in 2 ml increments until the peak viscosity break point was reached. Anhydrous sodium chloride was used when requirements exceeded 4% NaCl by weight. The results of these tests which report the viscosity at the break
35 point for each test solution and for each control are reported in the following Table 11.

TABLE II
VISCOSITY (CPS)

SURFACTANT= Na lauryl sulfate

Viscosity Builder	
N-dodecyl-2-pyrrolidone	12,285
coconut diethanol amide	13,000
lauryl dimethylamine oxide	10,140
cocamidopropyl betaine	17,850
cocamidopropyl hydroxy sultaine	12,425
Control	1,850

SURFACTANT= Na laureth sulfate (3 moles ethylene oxide)

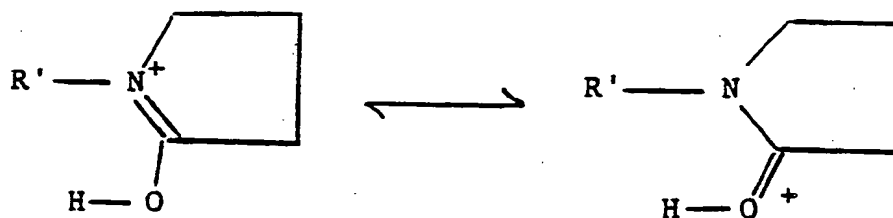
Viscosity Builder	
N-dodecyl-2-pyrrolidone	40,000
coconut diethanol amide	36,820
lauryl dimethylene oxide	31,590
cocamidopropyl betaine	55,680
cocamidopropyl hydroxy sultaine	54,540
Control	30,000

SURFACTANT= Ammonium lauryl sulfate

Viscosity Builder	
N-dodecyl-2-pyrrolidone	40,000
coconut diethanol amide	33,200
lauryl dimethylamine oxide	37,270
cocamidopropyl betaine	54,590
cocamidopropyl hydroxy sultaine	28,180
Control	6,360

EXAMPLE XXIXProtonation of N-n Alkyl Pyrrolidones to Form Cations

The cation formed by protonation of N-dodecyl pyrrolidone, i.e.



was precipitated from acid solution using a large anionic surfactant molecule, linear dodecyl benzene sulfonate.

10 Both the C₈ and C₁₀ alkyl pyrrolidones which are rather insoluble in water at pH 7, are readily solubilized in acid solution. Ten percent dispersions of the C₈ and C₁₀ pyrrolidones were titrated to a clear solution using HCl. The results are shown in the following table, Table 12.

TABLE 12

MOLAR RATIO OF HCl TO ALKYL PYRROLIDONE REQUIRED FOR
SOLUBILIZATION

N-n-alkyl group	Grams of Pyrrolidone (10% Dispersion)	HCl (ml.) 36.7%	Mole Ratio HCl/Pyrrolidone	Solution pH
octyl	20.0	5.2	6.2	0.22
decyl	20.0	9.7	12.8	<0.1

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To obtain a rough figure for the protonation equilibrium constant, the equilibrium [octyl pyrrolidone] was taken at 5×10^{-3} m. Additionally, the specific gravity of the 10% dispersion was taken at 1.0 g/cc. Calculation indicates that $K = 1.3 \times 10^2$ for the octyl pyrrolidone forming a cation, i.e. [octyl pyrrolidone] + $[H^+] \rightleftharpoons [\text{protonated pyrrolidone}]^+$.

Solubility of the present surfactants in highly alkaline solutions is limited and the pyrrolidone ring is not hydrolyzed in such environments. However the solubility in acid is very high. Data indicate that the present N-alkyl-2-pyrrolidones are much less prone to acid hydrolysis than N-methyl pyrrolidone. Mixtures (50/50) of the N-n alkyl pyrrolidones with 36.7% HCl were made. The C_8 and C_{10} alkyl pyrrolidones were clear, viscous liquids at 25°C., while the C_{12} alkyl/HCl mix was a homogeneous, transparent gel. The C_{14} /HCl (50/50) mixture became a non-homogeneous solid at room temperature.

EXAMPLE XXX

Anti-Corrosion Testing

The 9 N-alkyl-2-pyrrolidone products listed in the following table were tested in hydrochloric acid and contacted with oil well casing material to determine their efficacy as corrosion inhibitors in oil well acidizing.

Separate solutions each containing 0.4 g of the test compound in 100 mls of 15% active hydrochloric acid, were made up.

Steel Haliburton N-80 coupons used in oil well drilling were soaked in a 20% soap solution (Alkanox) for 20 minutes, then brush scrubbed with yellow soap and rinsed with water and then with acetone. The coupons were dried and weighed.

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After weighing, a coupon was placed in each of the beakers containing a test compound 20% HCl solution and warmed to 80°C. for 16 hours, after which the coupons were removed, rinsed, brush scrubbed with soap solution, dried and reweighed. The loss in weight of the coupon is recorded in following Table 13.

TABLE 13

<u>Steel Casing Corrosion Inhibitor Evaluation</u>		<u>Weight Loss (%)</u>
	Propargyl alcohol	0.21
10	N-decyl pyrrolidone (DP)	4.10
	N-octyl pyrrolidone (OP)	4.20
	N-dodecyl pyrrolidone (DDP)	5.95
	N-cyclohexyl pyrrolidone (CHP)	10.97
	N-methoxyethyl pyrrolidone (MEP)	14.16
15	N-methyl pyrrolidone (NMP)	14.55
	N-ethyl pyrrolidone (NEP)	14.56
	N-tetradecyl pyrrolidone (TDP)	14.82
	N-hydroxyethyl pyrrolidone (HEP)	15.57
	No inhibitor	27.2

Although propargyl alcohol showed the lowest weight loss, the present N-alkyl pyrrolidones present a viable alternative and are safer chemicals.

The above experiment was repeated except that the coupons were immersed in a 5% HCl solution for 16 hours. From this data, a corrosion inhibiting curve was plotted for the above inhibitors as shown in Figure 1 wherein % weight loss is plotted against the weight of the alkyl group. 2-Pyrrolidone (2P) was used for purposes of comparison by immersing a coupon for 16 hours at 80°C. in a 5% HCl solution containing 0.4 g of 2P. Interpolation of this curve indicates that N-nonyl-2-pyrrolidone is at least as effective as the N-octyl or the N-decyl species. N-tetradecyl pyrrolidone

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is a suitable corrosion inhibitor for weaker acids such as for example for preventing corrosion of dairy equipment in the presence of phosphoric acid cleaners; although, even in well acidization, it reduces metal corrosion by about 50%.

It is also found that the octyl, decyl, and dodecyl pyrrolidones are better inhibitors than cationic quaternary ammonium chloride in isopropanol such as the commercial corrosion inhibitor Katapone VV-328, which gave a 16% weight loss of the coupons in 15% HCl. Also this dark-colored quaternary ammonium chloride which is shipped as a 75% solution in isopropanol compares unfavorably with the present alkyl pyrrolidones which are water clear, 100% active and water soluble.

EXAMPLE XXXI

The density at 25°C., heat of vaporization in kilocalorie per mole and solubility parameter was determined for the C₈, C₁₀, C₁₂ and C₁₄ alkyl pyrrolidone species of the invention. These parameters are reported in following Table 14.

TABLE 14

PRODUCT	$d_{25^{\circ}\text{C}}$	Heat of Vaporizn. k cal/mole	Solubility Parameter (δ)
N-n-octyl-2-pyrrolidone	0.920	15.0	8.2
N-n-decyl-2-pyrrolidone	0.911	18.4	8.5
N-n-dodecyl-2-pyrrolidone	0.903	18.0	7.9
N-n-tetradecyl-2-pyrrolidone	0.896	19.7	7.8

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The present compounds possess dye transfer properties which permit better penetration of a dye into a cellulosic substrate. Accordingly, the present compounds are useful vehicles for dyeing or printing.

5

EXAMPLE XXXII

10

A 50% black dye formulation of 1:1.5 triarylmethane dye portion of C.I. Basic Blue and a methine dye portion of C.I. Basic Violet 16, C.I. Basic Yellow 29 and C.I. Basic Orange 21, dissolved in an aqueous vehicle containing 20 wt. % of N-dodecyl-2-pyrrolidone provides improved penetration of the dye into a swatch of white orlon fabric at room temperature and high exhaustion of dye on the fabric.

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Other representative formulations which suitably employ the products of this invention are presented below, although these are by no means limiting to the scope of suitable mixtures. The following formulations and compositions are prepared by conventional methods and require no detailed description. Generally the components are mixed at between about room temperature and about 100°C. under ambient pressure until a uniform composition is obtained.

Addition of the present lactams to any of the numerous commercial detergent and/or disinfectant solutions materially enhances their cleaning and sanitizing properties. The effective amount of lactam preferably employed ranges from about 0.2% to about 20% by volume of the total composition. The following formulations are representative.

A. Standard Dishwashing Compositions

	Vol. %
(i) Water	59.4
Ethanol (95%)	8.6
Alfonic 1412-A (59.3% (ethylene oxide sulfate)	22.5
Alfonic 1412-10 (linear alcohol ethoxylate)	1.1
Sodium Chloride	.9
N-decyl-2-pyrrolidone	7.5
	% by Wt.
(ii) Sulfated nonylphenoxypoly (ethyleneoxy)	
Ethanol ammonium salt	9.00
Cocamide diethanol amide	2.50
Ethoxylated nonylphenol (10 Mol. EO)	8.00
Nonylphenyl sulfonate (60%)	20.00
Sodium xylene sulfonate (40%)	9.00
50/50 mixture of N-n-octyl- and N-n-dodecyl-2-pyrrolidones	3.00
Fragrance	0.25
H ₂ O	48.25

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	B. <u>Machine Dishwashing Liquid</u>	% by Wt.
	Tetrasodium pyrophosphate	22.00
	Sodium metasilicate	10.00
	Sodium benzoate	1.00
5	Sodium xylene sulfonate (40%)	1.00
	Glycol ether	2.00
	Capryloamphocarboxy glycinate	6.00
	50/50 mixture of N-n-octyl- and N-n-dodecyl- 2-pyrrolidones	3.00
10	Fragrance	0.50
	H ₂ O	54.50
	C. <u>Bottle Washing Composition-Useful in pressure bottle washing equipment</u>	% by Wt.
	Cocoamphocarboxypropionate	1.00
15	Carbitol solvent (an alkyl ether of diethylene glycol)	1.00
	Sodium Hydroxide (flakes)	20.00
	N-dodecyl-2-pyrrolidone	5.00
	Water	73.00
20	D. <u>General Purpose Liquid Glass Cleaner</u>	% by Wt.
	Glycol ether (Arcosolve DPM)	4.00
	Ammonium Hydroxide (28%)	1.00
	Polyoxyethylene/polyoxypropylene block copolymer	0.10
25	N-n-Octyl-2-pyrrolidone	1.00
	Fragrance	Q.S.
	Water	to 100 %

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<u>E. Fine Fabric Washing Detergent</u>		% by Wt.
	Linear decyl benzene sulfonate	5.00
	Coconut diethanolamide	20.00
	Sodium lauryl ether sulfate (3 mol EO)	15.00
5	Sodium xylene sulfonate (40%)	10.00
	Citric Acid	to pH 7
	Preservative	Q.S.
	Colorant	Q.S.
	N-n-dodecyl-2-pyrrolidone	5.00
10	Water	to 100 %

Formulation E is particularly suited for woolen and nylon fabrics. The low surface tension of the pyrrolidone component permits high detergent action through the nap of knitted fabrics.

15	<u>F. Cold Water Phosphated Laundry Detergent</u>	% by Wt.
	Sodium tripolyphosphate	50.0
	Sodium silicate (2:1 ratio)	10.0
	Sodium sulfate	17.5
	N-C ₈ -C ₁₆ alkyl-2-pyrrolidone mixture	17.5
	H ₂ O	5.0

20 Grimey dirt and greasy sebum was easily removed by one cycle washing (15 min + 5 min rinse) with the above formulation at 80°F. The test was conducted in 120 ppm water hardness.

<u>G. Hard Surface Cleaners</u>		% by Wt.
25	(i) Tetrasodium phosphate	0.70
	Sodium metasilicate -5H ₂ O	0.50
	n-dodecyl benzene sulfonate	1.13
	N-n-octyl-2-pyrrolidone	0.75
	Sodium xylene sulfonate	6.80
30	H ₂ O	90.12

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The improved cleaning capacity of this formulation is attributed to the co-surfactant function of n-octyl pyrrolidone with n-dodecyl benzene sulfonate.

(ii)

5	Sodium lauryl ether sulfate (3 Mol EO)	20.00
	Coconut diethanolamide	10.00
	Ethylene glycol monobutyl ether	5.00
	Tetrasodium ethylene diamine tetraacetic acid (EDTA)	1.00
10	50/50 mixture of N-n-octyl- and N-n-dodecyl-2-pyrrolidones	3.00
	Fragrance	Q.S.
	Preservative	Q.S.
	Colorant	Q.S.
15	Water	to 100%

H. Disinfectant; Sanitizing & Decontaminating Detergents

(i)

% by Wt.

	Bardac 205M (octyldecyl dimethyl benzyl ammonium chloride)	2.80
20	N-dodecyl-2-pyrrolidone	1.20
	H ₂ O	96.00

(ii)

	Miranol C 2M-SF (dicarboxylic coconut derivative sodium salt, amphoteric)	15.00
25	Quaternary ammonium salt, 50% (Decyldimethyl octyl ammonium chloride)	2.00
	Sodium carbonate	2.00
	Ethylene tetraacetic acid	0.50
	N-n-dodecyl-2-pyrrolidone	0.50
30	H ₂ O	80.00

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(iii)

	Benzalkonium chloride	5.00
	Sodium carbonate	2.00
	Sodium citrate	1.50
5	Nonoxynol 10 (10 av. ethoxylated nonyl phenol)	2.50
	N-n-octyl-2-pyrrolidone	5.00
	H ₂ O	84.00

(iv)

% by Wt.

	Lauric/myristic diethanolamide	7.00
10	Sodium lauryl sulfate	6.00
	Trisodium phosphate	2.00
	Sodium tripolyphosphate	2.00
	N-n-octyl-2-pyrrolidone	5.00
	Fragrance	0.50
15	H ₂ O	77.50

(v)

% by Wt.

	Magnesium aluminium silicate	0.90
	Kelzan gum thickener	0.45
	tetrasodium EDTA	1.00
20	Monazoline-O [*] /Imidazoline	1.00
	Hydrochloric acid (37%)	20.00
	Barquat MB-80 (alkyl dimethyl benzyl ammonium chloride)	1.25
	50/50 mixture of N-n-octyl- and N-n-dodecyl-2-pyrrolidones	3.00
25	Fragrance (acid stable)	1.00
	H ₂ O	71.40

* substituted imidazoline of oleic acid

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The formulations H-(i) through H-(v) are particularly suited for hospital and institutional use in washing porcelain tile, tubs, toilet bowls, sinks, shower stalls, etc., associated fixtures and floors. They are also effective cleaning liquids which reduce or eliminate animal odors as may be encountered in a veterinarian hospital or doctor's office or in the home, since the N-alkyl lactam possesses the property of complexing with urea and mercapto type compounds. For odor masking effects somewhat higher amounts of the pyrrolidone component, eg. up to about 10% of the formulation, may be employed, if desired.

I. General Purpose Medium Duty Liquid Alkaline Cleaner

		% by Wt.
	Sodium hydroxide (50%)	1.00
15	Potassium hydroxide (45%)	1.50
	Sodium metasilicate (anhydrous)	2.50
	Sodium tri-polyphosphate	3.00
	Nitrilotriacetic acid	3.00
	Monateric CEM-38 (Coconut amphoteric surfactant)	2.00
20	Monafax 831 (a phosphate ester)	1.00
	50/50 mixture of N-n-octyl- and N-n-dodecyl-2-pyrrolidones	3.00
	Fragrance	0.50
25	H ₂ O	82.50

Formulation I is particularly useful for cleaning metal surfaces, ceramic tile and household appliances.

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J. <u>Dairy Equipment Liquid Cleaner</u>		% by Wt.
	Gluconic Acid (50%)	20.00
	Sodium Nonoxynol -9 phosphate*	10.00
	N-n-octyl-2-pyrrolidone	5.00
5	H ₂ O	65.00

* the sodium phosphate of 9 av. ethoxylated nonyl phenol

K. <u>Leather, Vinyl and Other Plastic Liquid Cleaner</u>		% by Wt.
	Ethoxylated alkylphenol	10.00
	Arcosolve PM (propylene glycol methyl ether)	5.00
10	Isopropyl alcohol	2.50
	Amyl acetate	1.00
	50/50 mixture of N-n-octyl- and N-n-dodecyl-2-pyrrolidones	2.00
	Fragrance	Q.S.
15	Preservative	Q.S.
	H ₂ O	to 100%

L. <u>Liquid Rug Shampoo</u>		% by Wt.
	Sipex 7WC concentrate (blend of ionic and nonionic surfactants, C ₁₂ av. chain length)	10.00
20	Lauryl ether sulfate (3 mole EO)	10.00
	Sodium tripolyphosphate	2.00
	Ethyl carbitol solvent	1.50
	Tinopal 5BM optical brightener (diamino stilbene)	0.05
25	N-n-dodecyl-2-pyrrolidone	2.00
	Fragrance	Q.S.
	Preservative	Q.S.
	H ₂ O	to 100%

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The complexing properties of the pyrrolidone component, as explained in the formulations H-(i) through H-(v), enable the shampoo formulation to eliminate pet odors as well as providing superior cleaning and depositing a microfilm of pyrrolidone which acts as a barrier against redeposit of soil.

5	<u>M. Detergent Rinse Aid</u>	% by Wt.
	Nonoxynol 9 (9 av. ethoxylated	
	nonyl phenol)	30.00
10	Isopropanol	15.00
	Propylene glycol	15.00
	N-n-octyl-2-pyrrolidone	3.00
	H ₂ O	37.00

15 Addition of the pyrrolidone component in the above formulation significantly reduces the rinsing time to more effectively and completely remove soapy deposits; thus preserving color brightness.

20	<u>N. Fabric Softener</u>	% by Wt.
	Miranol DM (monocarboxylic stearic derivative, sodium salt)	3.00
	Arquad 2HT 75 (dimethyl[hydrogenated tallow] ammonium chloride)	2.00
	N-n-dodecyl-2-pyrrolidone	1.00
	Fragrance	0.25
25	H ₂ O	93.75

Formulation N can be added directly to the washing detergent or used to impregnate non-woven strips employed in a clothes dryer or sprayed directly on fabric after laundering.

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O. Antistat formulations

(wash cycle additive)

% by Wt.

	(i) Sodium silicate (2:1 ratio SiO_2 to Na_2O)	10.00
	Lauric acid	0.20
5	Sodium hydroxide	0.20
	Sodium carbonate	33.25
	Sodium sulfate	43.20
	(1) Igepal CO-630	7.00
	N-n-dodecylpyrrolidone	5.00
10	H_2O	1.15

(1) 100% active liquid condensation product of nonyl alcohol and nine units of ethylene oxide

15 The above formulation significantly lowered the static electricity on clothes dried in an automatic dryer and is compatable with wash cycle detergents.

20 (ii) Melted polypropylene having a melt flow index of 8 and a density of 0.902 at 200°C. was mixed with 1% by weight of N-dodecyl-2-pyrrolidone and extruded from a twin screw extruder to form bottles. The same operation was repeated with the melted polypropylene, but omitting the 1% pyrrolidone. Unlike the pyrrolidone deficient composition, those bottles containing pyrrolidone did not pick up dust or lint after 10 passes over a nylon cloth and their surface resistivity was measured at 1×10^{12} ohm

25 at 30% humidity, as opposed to 4×10^{14} ohm (30% humidity) reported for the pyrrolidone defficient polypropylene bottles.

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P. Liquid Softening/Antistat Composition % by Wt.

	N-n-tetradecylpyrrolidone	5.4
	(2) Igepal CO-660	23.9
	H ₂ O	55.7
5	Ethanol	15.0

(2) 100% active liquid/liquid condensation product of nonyl alcohol and ten units of ethylene oxide

The above formulation exhibited the same properties as Formulation C.

10 Antistatic compositions incorporating from about 5% to about 100% of the present lactams as the active ingredient are also usefully applied as aerosol sprays to rugs, clothing and furniture whereby the anionic charges accumulated on these surfaces are neutralized and

15 discharged by the mildly cationic character of the present lactams below pH 7.

Q. Complexed Compositions % by Wt.

	H ₂ O	60.99
	N-n-dodecylpyrrolidone	10.48
20	Sodium iodide	22.20
	Iodine	6.33

The N-n-dodecyl pyrrolidone-iodine complex (iodophor) which formed provides improved iodine solubility for disinfecting purposes.

25	R. <u>Acid thickener</u>	% by Wt.
	Concentrated HCl	50.0
	N-n-dodecylpyrrolidone	50.0

30 The H⁺ complexation of the N-n-dodecyl pyrrolidone with the acid significantly increased the acid viscosity so that the formulation can be applied to vertical surfaces without runoff.

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S. SKIN LOTION

	<u>Ingredient</u>	<u>% by Wt.</u>
	Stearic Acid	3.00
	Mineral Oil, 70 cts	2.00
5	Emulsifying Wax	3.00
	Dimethicone	1.50
	Deionized Water	QS
	Carbomer 934 *	0.15
	Oleth-20 **	1.00
10	N-decyl-2-pyrrolidone	1.00
	Triethanolamine, 98%	1.00
	Preservative	QS
	Fragrance	QS

T. FACIAL CREAM

	<u>Ingredient</u>	<u>% by Wt.</u>
15	Mineral Oil, 70 cts	6.00
	Petrolatum	4.00
	Lanolin	3.00
	Glyceryl Monostearate,	
20	S.E. Acid Stable	19.00
	Glycerine	1.00
	N-octyl-2-pyrrolidone	2.00
	Deionized Water	QS
	Preservative	QS
25	Fragrance	QS

* a crosslinked polymer of acrylic acid (B.F. Goodrich)

** the polyethylene glycol ether of oleyl alcohol
(GAF Corp.)

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U. SUNSCREEN LOTION

	<u>Ingredient</u>	% by Wt.
	Myristyl Myristate	1.00
	PVP/Eicosene Copolymer	2.00
5	Glyceryl Stearate, S.E.	3.50
	Dimethicone	1.00
	N-dodecyl-2-pyrrolidone	2.00
	Deionized water	Q.S.
	Carbomer 940	0.10
10	Triethanolamine	0.10
	Preservative (Germaben II) *	Q.S.
	Octyldimethyl p-aminobenzoic acid	4.00
	Fragrance	Q.S.

V. HAIR SHAMPOO

	<u>Ingredient</u>	% by Wt.
15	C ₁₄ -C ₁₆ Alpha Olefin Sulfonate	20.00
	Ammonium Lauryl Sulfate	25.00
	Cocamidopropyl Betaine	3.50
	N-dodecyl-2-pyrrolidone	1.00
20	Sodium Laureth-4-Phosphate	1.00
	Hydrolyzed Animal Protein	0.25
	Tetrasodium ethylene diamine tetra acetic acid	0.15
	Deionized water	Q.S.
25	Fragrance	Q.S.
	Preservative (Kathon CG) **	Q.S.

* N-[1,3-bis(hydroxymethyl)-2,5-dioxo-4-imidazolidinyl-N,N'-bis(hydroxymethyl)urea; Sutton Labs.

** 5-chloro-2-methyl-4-isothiazolin-3-one (Rohm & Haas)

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W. BODY SHAMPOO

	<u>Ingredient</u>	% by wt.
	Deionized water	Q.S.
	C ₁₄ -C ₁₆ Alpha Olefin Sulfonate	35.00
5	Sodium Methyl Cocyl Taurate	12.00
	Polyethylene glycol-150 Distearate	1.00
	Glycol Distearate	2.00
	N-dodecyl-2-pyrrolidone	6.00
	Polyquaternium-11	2.50
10	Cocamidopropyl Betaine	10.00
	Citric Acid	to pH 6.0
	Fragrance	Q.S.
	Preservative (Kathon CG)	Q.S.

X. BRUSHLESS SHAVING CREAM

	<u>Ingredient</u>	% by Wt.
15	A. Stearic Acid	20.00
	Cetyl Alcohol	1.00
	Lanolin	2.00
	Isopropyl Palmitate	6.00
20	(Part A added molten and mixed with Part B at 80°C.)	
	B. Hexylene Glycol	8.00
	Triethanol Amine	1.80
	Potassium Hydroxide	0.50
	Borax	2.00
25	N-tetradecyl-2-pyrrolidone	2.00
	Deionized water	Q.S.
	Preservative (Kathon CG)	Q.S.
	Fragrance	Q.S.

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Y. AEROSOL SHAVING CREAM

	<u>Ingredient</u>	<u>% by Wt.</u>
	Deionized Water	Q.S.
	Glycerine	5.8
5	Oleth-20	1.0
	Butylated hydroxy anisole	0.1
	Butylated hydroxy toluene	0.1
	Stearic Acid	7.5
	Lanolin	0.5
10	Mineral Oil, 70 cts	2.4
	Cetyl Alcohol	0.5
	Triethanolamine, 98%	3.9
	Cocamide Diethanolamine	0.5
	N-dodecyl-2-pyrrolidone	2.0
15	Fragrance	0.5

Concentrate : Propellant Ratio - 95:5

Propellant : A-46 [80:20 - Isobutane/Propane]

20 The incorporation of the present lactams in any of the cosmetic formulations provides additional moisturizing and softness to the skin and hair as well as superior penetration and promotes creamy smooth emulsions and/or thicker consistency to any of the cream or lotion formulations.

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Z. Hand Lotion

Parts by Wt.

Water	81
Propylene glycol	2
Petrolatum	3
Stearic acid	6
Triethanolamine	1
Glycerin	2
N-dodecyl-2-pyrrolidone	5

AA. Waterless Hand Cleaner

% by Wt.

10	Deionized Kerosene	44.00
	Stearic acid	4.00
	Ethoxylated nonyl phenol	4.00
	Propylene glycol	4.00
15	Arcosolve DPM (dipropylene glycol mono-methyl ether)	3.00
	Triethanolamine	1.00
	N-n-octyl-2-pyrrolidone	3.00
	Fragrance	Q.S.
	Preservative	Q.S.
20	H ₂ O	Up to 100%

The solubilizing effect of the pyrrolidone on grease, oil and tar deposits, coupled with improved skin penetrating capability, contribute to the superior cleaning power of formulation AA.

25 BB. Cold Cream

Parts by Wt.

Water	58
Propylene glycol monostearate	4
Lanolin	6
Mineral Oil	26
Triethanolamine	1.5
N-nonyl-2-pyrrolidone	4.5

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CC. <u>Insect Repellant Gel</u>		Parts by Wt.
	Metadelphene	600
	Ethanol	100
	Carboxymethylene	10
5	N-tallow-2-pyrrolidone	15
	Triethanolamine	8

DD. <u>Aerosol Pesticidal Formulation</u>		% by Wt.
	Dowicide 1 (2-phenylphenol)	5.00
10	Glycol ether of Dowanol DPM (Dipropylene glycol monomethyl ether)	10.00
	Deionized kerosene	10.00
	Aerothene TT solvent (1,1,1-trichloroethane)	40.00
15	Propellant A-46 (⁸⁰ / ₂₀ vol. mixture of isobutane and propane)	25.00
	N-n-dodecyl-2-pyrrolidone	10.00

Formulation DD is particularly suited for use against flies, mosquitoes and ticks and may be safely sprayed on animals, humans and inanimate substrates to kill or deter attack by such pests.

EE. Toxicity Reducing Formulation

Ingredient		% by Wt.
25	Aldicarb	7.5
	N-alkyl pyrrolidone mixture: C ₈ , C ₁₂ and C ₁₈ alkyl in ratio 5:2.5:2.5	2.0%
	Igepon T 77	11.0%
	Xylene	3.0%
30	H ₂ O	76.5%

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The toxicity of Aldicarb, i.e. 2-methyl-2-(methylthio) propanal O-[(methylamino)carbonyl] oxime, in the above formulation is reduced by at least $1/10$ over that in which no lactams was employed and the formulation EE provides high mortality to cockroaches, ants and other pests over an extended period of time.

FF. Herbicidal and Fungicidal Adjuvant Formulation

The following formulation has been specially developed for triazine herbicides in post emergent applications in order to increase the application rate without reducing weed control and, at the same time, reducing harmful residues, thus allowing quick crop rotation.

The present N-alkyl lactams can be built into a suspension concentrate without detracting from the physical stability of the product.

A concentrate formulation

Atrazine (2-chloro-4-ethylamino-6-isopropyl-amino,-1,3,5-triazine)	250 grams
N-n-octyl-2-pyrrolidone	328 grams
H ₂ O	to 1 liter

GG. Emulsifiable Concentrate Formulations for Agricultural Chemicals

(i) The following formulations (1-5) describe herbicidal 2,4-D (isopropyl ester of 2,4-dichlorophenoxy acetic acid) emulsifiable concentrates in various solvents which contain a 50/50 wt % mixture of N-dodecyl-2-pyrrolidone and Emulphor EL-620 and which, when added to water, produce stable emulsions.

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#	herbicide % by Wt.	Surfactant Blend % by Wt.	Xylene % by Wt.	Kerosene % by Wt.	Velsicol* AR-50 % by Wt.	Shell** F-407-R % by Wt.
1	39	5	-	56	-	1
2	44	5	51	-	-	-
3	44	5	-	-	51	-
4	40	5	-	-	21	-
5	47	5	-	-	-	48

The same formulations can be used for the butyl and other alkyl esters of 2,4-D. The addition of the pyrrolidone mixture in the above formulation controls viscosity, provides a stable emulsion and better distribution of the formulation on the vegetation.

(ii) The following formulations (1-7) describe suitable insecticidal (chlordane) emulsifiable concentrates containing a 10/40 wt. % blend of N-n-octyl-2-pyrrolidone and Emulphor EL-620 optionally combined with varying amounts of Igepal CO-630. The Igepal containing blends when added to water produce fast breaking emulsions; whereas those omitting Igepal are stable.

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	#	Chlordane* wt. %	Igepal CO-630 wt. %	Pyrrolidone/ Emulphor Blend wt. %	Rutyl Cellosolve wt. %	Kerosene wt. %
5	1	50	-	35	-	-
	2	50	-	35	15	-
	3	46	2	-	-	46
	4	46	2.5	-	-	49
	5	75	-	5	-	20
10	6	75	10	-	-	15
	7	46	9	-	-	45

* 1,2,4,5,6,7,8,8-Octachloro-2,3,3a,4,7,7a-hexahydro-4,7-methanoindane

15 (iii) The following formulations (1-4) describe suitable insecticidal (toxaphene) emulsifiable concentrates containing a 20/20/40 blend of N-n-decyl-2-pyrrolidone, N-n-dodecyl-2-pyrrolidone and Emulphor EL-620, optionally combined with varying amounts of Igepal CO-530. The

20 Igepal containing blends, when added to water provide fast breaking emulsions; whereas those which omit Igepal provide stable emulsions.

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#	Toxaphene* wt. %	Igepal CO-530 wt. %	Surfactant Blend wt. %	Kerosene wt. %	Butyl Cellosolve wt. %
1	50	15	35	-	-
5	2	50	-	35	-
3	45	8	-	47	-
4	45	4	-	50	-

* chlorinated camphene

10 The pyrrolidone in the toxaphene formulation provides the same promotional effect as noted for chlordane.

HH.	<u>Herbicideal Wettable Powder Formulations</u>	% by Wt.
(i)	Isopropylphenyl carbamate	50.00
	Hi-Sil (hydrated amorphorous silica)	46.00
15	Marasperse N (sodium lignosulfonate)	2.00
	N-n-octyl-2-pyrrolidone	2.00

Above formulation has excellent suspension and dispersion in hard and soft water.

(ii)		% by Wt.
20	Chlordane	40.00
	Attaclay (attapulgate)	55.00
	Blancol	3.00
	N-n-octyl-2-pyrrolidone	2.00
(iii)		% by Wt.
25	Chlordane	40.00
	Attaclay	56.50
	Marasperse N	2.00
	Igepon T 77	0.50
	N-n-dodecyl-2-pyrrolidone	1.00

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	(iv)		% by Wt.
		Toxaphene	40.00
		Attaclay	56.00
5		Daxad 27 (Na salt of a polymerized substituted benzoid alkyl sulfonic acid)	3.00
		N-n-octyl-2-pyrrolidone	1.00
	(v)		% by Wt.
		Toxaphene	40.00
10		Attaclay	55.00
		Blancol	4.00
		N-n-octyl-2-pyrrolidone	1.00
	(vi)		% by Wt.
		Toxaphene	40.00
15		Attaclay	55.00
		Marasperse N	4.00
		N-n-octyl-2-pyrrolidone/N-n-dodecyl- 2-pyrrolidone 75/25 mixture	1.00
	(vii)		% by Wt.
20		Aldrin (1,2,3,4,10,10-hexachloro-1,4,4a, 5,8,8a-hexahydro-exo-1,4-endo-5,8- dimethano-naphthalene	25.00
		Attaclay	71.50
		Blancol	2.00
25		Igepon T-77	0.50
		N-n-octyl-2-pyrrolidone	1.00
	(viii)		% by Wt.
		Aldrin	50.00
		Attaclay	45.00
30		Marasperse N	3.00
		N-n-decyl-2-pyrrolidone	2.00

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(ix)		% by Wt.
	Aldrin	75.00
	Hi-Sil	20.00
	Blancol	3.00
5	N-n-dodecyl-2-pyrrolidone	2.00

(x)		% by Wt.
	Dieldrin (Hexachloro-epoxy-octahydro- endo, exo-dimethanonaphthalene)	25.00
	Attaclay	71.50
10	Blancol	2.00
	Igepon T-77	0.50
	N-n-octyl-2-pyrrolidone	1.00

(xi)		% by Wt.
	Dieldrin	50.00
15	Attaclay	45.00
	Blancol	3.00
	N-n-dodecyl-2-pyrrolidone	2.00

The above formulations are milled on a fly cutter mill at 20,000 rpm for 1 minute at room temperature and provide concentrates having a wetting time less than 30 seconds.

II. <u>Fungicidal Wettable Powder Formulation</u>	% by Wt.
Phenyl Mercuric Acetate	90.00
N-n-octyl-2-pyrrolidone	1.00

The formulation provides a free flowing, non-bleeding powder; however only 0.1 to about 0.5% by weigh of N-n-octyl-2-pyrrolidone is required to impart good wettability to this fungicidal powder.

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JJ. Paint Remover & Paint Brush Cleaner Parts

Benzene	4
Fusel Oil	3
Alcohol	1
N-hexadecyl-2-pyrrolidone	10

5

KK. Rodent Paste Parts

Lard	1
Flour	1
White Arsenic	2
N-eicosyl-2-pyrrolidone	2
Oil of anise	10 drops

10

LL. Rust Remover Wt. %

GAFTEX PT (methyl vinyl ether/ maleic anhydride copolymer)	7
Citric Acid	5
Water	63
N-cocoalkyl-2-pyrrolidone	20
Polyvinyl pyrrolidone K90	5

15

20

The lactam mixture forms a coating over the metal, thereby inhibiting subsequent rust formation on the cleaned surface.

MM. Perfume Parts by Wt.

Lilac	80
Muguet	5
3% Musk extract	5
Tuberose absolute	2
Jasmine	8
90% alcohol	800
N-pentadecyl-2-pyrrolidone	100

25

30

Retention of the perfume odor on the skin is remarkably extended.

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NN. <u>Spray-Wipe Furniture Polish</u>		% by Wt.
	Petrolite C-36 emulsion*(20%)	3.50
	Isopar E solvent (C ₈ -C ₉ isoparaffin mixture of branched chain aliphatic hydrocarbons)	32.50
5	S-Maz 80 (Sorbitan monooleate)	0.20
	Masil EM 1000 emulsion (dimethyl polysiloxane silicone emulsion, 60% active)	3.40
10	50/50 mixture of N-n-octyl- and N-n-dodecyl-2-pyrrolidones	3.00
	Fragrance	0.40
	H ₂ O	57.00

*reacted microcrystalline wax, m.p. 195°F, needle penetration at 77°F is 7.5

The use of the present lactams in any of the commercially available furniture and metal polishes promotes cleaning as the lactam exerts its complexability with soil, grease or oily deposits, thus facilitating their removal. Furniture polish formulation NN also provides superior penetration into the wood surface and diffuses through previous wax deposits.

OO. <u>Dry Cleaning Formulations</u>		% by Wt.
25	(i) Perchloroethylene	94.50
	Isopropyl methyl cellulose	0.50
	N-octyl-2-pyrrolidone	5.00
30	(ii) Perchloroethylene	46.00
	Ethoxylated nonylphenol phosphate ester (GAFAC RS-610 and PE-510, 2:1)	31.00
	N-octyl-2-pyrrolidone	15.00
	Potassium hydroxide	8.00

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	(iii)	Stoddard solvent (a petroleum distillate between gasoline & kerosene)	% by Wt.
			41.50
		GAFAC RS-610	30.00
5		Nekal WT-27 (sulfonated aliphatic polyester)	7.50
		Hexylene glycol	10.00
		Potassium hydroxide	8.00
		N-dodecyl-2-pyrrolidone	3.00
10	(iv)	Perchloroethylene	40.00
		Isopropanol	5.00
		N-dodecyl-2-pyrrolidone	50.00
		Water	5.00

15 The above formulations are highly effective in removal of wine, tea, fruit and other water soluble and oil soluble stains together with normal soil. Additionally, the present lactams eliminate any perspiration stains or body odor remaining in a fabric.

20 The above and many other formulations which require one or more of the properties imparted by the present pyrrolidone products are suitably employed in the present invention. Also, any of the N-C₇ to C₂₂ alkyl-caprolactams can be substituted therein to provide those benefits described for the alkyl pyrrolidones.

25	PP.	<u>Decontaminating Solution For Removal Of</u>	% by Wt.
		<u>Radioactive Material</u>	
		Sodium lauryl ether sulfate.	10.00
		N-dodecyl-2-pyrrolidone	5.00
		Cocoamidopropylbetaine	10.00
30		H ₂ O	to 100%

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Formulation PP is effective in removing contamination from surfaces, eg. radioactive contaminated surfaces encountered in a diagnostic laboratory. Also radioactive iodine, in concentrations as low as parts per billion, can be completely washed off individuals quickly and efficiently with the above formulation. When the pyrrolidone component is omitted, however, the residual iodine remains on the skin.

10	QQ. <u>Formulation for Industrial Odor Control</u>	% by Wt.
	N-octyl-2-pyrrolidone	2.00
	Dodecylbenzene sulfonic acid	5.00
	Sodium xylene sulfonate	2.00
	Ethoxylated (av.9) nonylphenol	5.00
	H ₂ O	to 100%

15 Formulation QQ was sprayed at a rate of 1 lb./cubic yard over the soil in which odoriferous lauryl mercaptan had been accidentally spilled. After 1 hour, the objectionable odor was completely eliminated.

20	RR. <u>Odor Removing Rug Shampoo</u>	% by Wt.
	Sodium lauryl sulfate	12.00
	N-dodecyl-2-pyrrolidone	3.00
	Sodium xylene sulfonate	2.00
	H ₂ O	to 100%

25 Formulation RR was applied to a new rug which had been soiled by dog urine and had a strong amine/mercaptan odor. After drying, the odor of treated rug was reduced to the mildest trace. The application can be repeated for complete odor removal. The omission of pyrrolidone in the above formulation had no odor removing effect whatever
30 when applied to another portion of the rug soiled in the same manner.

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SS.

LIQUID METAL POLISH

	<u>Ingredient</u>	<u>% by Wt.</u>
	2-heptadecyl-1-carboxymethyl-1-(2-hydroxyethyl)-	
5	2-imidazolinium chloride	10.00
	Xanthan gum	0.30
	Magnesium aluminum silicate	2.00
	Hydrated amorphous silica	12.50
	N-n-octyl-2-pyrrolidone	5.00
10	Fragrance	0.50
	Water	<u>Q.S.</u>
		100.00

The above formulation is particularly useful on oxidized metals, eg. silver, wherein the oxide is complexed with the lactam and easily removed from the metal surface.

15 TT.

LIQUID BLACK SHOE POLISH

	<u>Ingredient</u>	<u>% by Wt.</u>
	Carnauba wax, 82.5°-86.0°C. (melting point)	4.00
	Dimethyl polysiloxane, 200 CPS	3.00
20	Nonionic polypropylene glycol	1.00
	(C-18) Fatty acid ester polyglyceryl-4-oleate	0.70
	Carbon black (10% dispersion in C ₈₋₉ Isoparafin)	4.00
	C ₈₋₉ Isoparafin	20.00
25	N-dodecyl-2-pyrrolidone	5.00
	Water	Q.S.
	Preservative	<u>Q.S.</u>
		100.00

The above shoe polish moisturizes and conditions the leather while providing good shine.

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UU.

(HARD STONE) JEWELRY CLEANER

	<u>Ingredient</u>	<u>% by Wt.</u>
	Ammonia, 28° baumé	10.00
5	Ethoxylated alkyl phenol (Igepal CA-630)	1.00
	Alkali stable dye	Q.S.
	N-octyl pyrrolidone	3.00
	Water, Deionized	to 100%

10 Formulation NN is particularly good for cleaning non-porous stones such as diamonds, rubies, emeralds, sapphires, topaz, zircons, etc. as well as gold, silver and platinum mountings. Greasy and soapy deposits are effectively complexed with the lactam and removed.

15 The present N-alkyl lactams are also excellent complexing agents as shown in the following Examples.

EXAMPLE XXXIII

20 In a glass round bottom 100 ml flask equipped with a thermometer and a condenser was added 24.34 g (0.1 mole) of N-dodecyl-2-pyrrolidone and 11.1 g (0.1 mole) hydroquinone. The mixture was heated and stirred until a homogeneous liquid phase was obtained. The melt was maintained at about 135°C. for 30 minutes, whereupon it was chilled to about 20°C. The crystalline solid product which formed had a m.p. of 78-82°C. The complex structure was evidenced by IR spectra which showed a shift of the pyrrolidone carbonyl from 1692 CM^{-1} to 1654 CM^{-1} and 1612 CM^{-1} . Proton nuclear magnetic resonance ($^1\text{H NMR}$) at 25° \pm 2°C. indicated a 1:1 molar complex.

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EXAMPLE XXXIV

Example XXXIII is repeated except that only 12.17 g (0.05 mole) of N-dodecyl-2-pyrrolidone is used and 13.32 g (0.05 mole) of pentachlorophenol is substituted for 11.1 g of hydroquinone and the resulting melt is heated at 120°C. for 30 minutes. A solid crystalline complex of 1:1 molar N-dodecyl-2-pyrrolidone/pentachlorophenol is formed.

EXAMPLE XXXV

Example XXXIV is repeated except that 7.61 g (0.05 mole) of vanillin was substituted for 13.32 g of pentachlorophenol. The mixture was heated until homogeneous and was then maintained at 60°C. for 30 minutes. A low melting crystalline complex was formed.

EXAMPLE XXXVI

In an NMR tube, a mixture of 0.1 molar N-dodecyl-2-pyrrolidone and different concentrations of phenol, from 0.1 to 0.3 M, was dissolved in deuterated chloroform and analyzed by 300 mega hertz ¹HNMR at 25 ± 2°C. In each case, the phenolic OH shifted to higher frequency in the presence of N-dodecyl-2-pyrrolidone which shows complexation of N-dodecyl-2-pyrrolidone and phenol through intermolecular hydrogen bonding.

EXAMPLE XXXVII

To 200 g of beer is added 50 g of N-n-dodecyl-2-pyrrolidone with stirring until a uniform mixture is obtained and the mixture is cooled to 15-20°C. to provide a clear solution. The solution is then heated to 25°C. whereupon two liquid phases are formed and separated.

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5 The clear beer fraction, which is the lower phase, is recovered and analyzed by the method of J. Jerumanis (Brauwissenschaft Vol. 25, #10, pages 319-321, 1972). Analysis shows that 90% of the phenolic impurities are removed.

EXAMPLE XXXVIII

10 To 200g. of beer is added 50g. of N-octadecyl-2-pyrrolidone with stirring for 30 minutes at 25°C. The beer is then filtered to remove the N-octadecyl pyrrolidone/polyphenolic complex which was formed. Analysis by the method of the preceding example shows that 85% of the polyphenols are removed.

EXAMPLE XXXIX

15 A 2x2 inch swatch of white 100% cotton was impregnated with blue black ink in a 0.5 inch circular area. The swatch was then held under hot water while rubbing for 1 minute. A barely noticeable amount of the ink was removed.

20 The swatch was then placed on a clean counter and 1 drop of 100% N-n-dodecyl-2-pyrrolidone from a medicine dropper was contacted with the ink spot, and allowed to remain for 1 minute; after which the swatch was held under warm water while rubbing for 0.5 minute. The sample was then dried and examined for ink removal. Only the
25 faintest shadow, barely discernable, blue color remained.

The above experiment was repeated except that 100% N-n-octyl-2-pyrrolidone was substituted for N-n-dodecyl-2-pyrrolidone. Dye removal was even more complete so that magnification was needed to detect color.

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The above compounds can be employed individually or in admixture and used as the sole spot removing agent for home or commercial laundering or in a dry cleaning operation. The compounds may also be incorporated into a commercial stain and spot removing formulation to boost stain removing properties.

EXAMPLE XL

A mixture of glycerine containing about 0.2% N-n-dodecyl-2-pyrrolidone was applied morning and evening to the entire finger nail surface and around the nail bed cuticle of subject A. The nails of subject A were in poor condition, i.e. striated and subject to breakage, splitting and peeling. The above treatment was repeated for 10 consecutive days. Two weeks after the treatment was discontinued, the nails were examined. Resistance to breakage was remarkably improved. Splitting and peeling were also noticeably reduced. Nail striation was not diminished, however the nails and cuticle appeared less dry and striations less noticeable.

EXAMPLE XLI

To improve the dissolution rate of hydrochlorothiazide tablets, the blend:

	% by Wt.	Per tablet
Hydrochlorothiazide	10.0	45.00 mg
N-octyl-2-pyrrolidone	0.5	2.25 mg
1:1 mixture of lactose:		
dicalcium phosphate	88.5	398.25 mg
Magnesium stearate	1.0	4.50 mg

was prepared by blending the hydrochlorothiazide with an ethanolic solution of the N-octyl-pyrrolidone and dried in

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an oven at 60°C. The dried material was blended with the lactose/dicalcium phosphate diluent for 7 minutes, after which the magnesium stearate lubricant was added and mixed for an additional 3 minutes. The resulting material was then compressed into tablets of 8-10 Kp hardness by using a Stokes B-2 tablet press (Sample B).

Alternatively, the above procedure was repeated except that the ethanolic pyrrolidone solution was added to preblended hydrochlorothiazide and diluent (Sample C).

The above procedure was repeated except that the addition of N-octyl pyrrolidone was omitted to provide a control formulation (Sample A).

Finally the above procedure was again repeated, except that the conventional sodium lauryl sulfate surfactant was substituted for the N-octyl-pyrrolidone surfactant to provide a standard against which the efficacy of the present lactam was measured (Sample D).

The dissolution of Hydrochlorothiazide was then measured for Samples A, B, C and D using the USP method and results of these measurements reported in following Table I.

TABLE I

	%	%	%	%
Sample	after 30 min	after 60 min	after 90 min	after 120 min
A	7.93	20.80	33.46	44.80
B	26.20	67.03	90.48	95.00
C	----	----	----	98.00
D	----	----	----	100.00

The N-octyl pyrrolidone significantly enhanced the dissolution rate of the drug from a directly compressible system and proved to be almost as effective as sodium

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lauryl sulfate while superior in other properties such as its non-irritatability and non reactivity with acidic drugs and pharmaceutical excipients. It is well known that sodium lauryl sulfate is highly irritating and its alkaline character causes it to react with acidic components which leads to compatibility problems as well as problems with the physical characteristics of the finished products.

The above procedure for preparing Sample B was twice repeated except that 0.1% by weight and 0.5% by weight of N-dodecyl-2-pyrrolidone was substituted for 0.5% by wt of N-octyl-2-pyrrolidone and the balance of the formulation was taken up by the diluent (Samples E and F respectively).

The dissolution rates were measured, the results of which are reported in following Table II.

TABLE II
% Dissolution of Drug

Sample	After 30 min	After 60 min	After 90 min	After 120 min
E (0.1% NDP)	9.63	27.24	49.64	67.64
F (0.5% NDP)	19.92	59.11	87.07	96.20

The procedure for preparing Sample B was repeated 4 times except that 0.5 wt % of N-decyl-2-pyrrolidone (Sample G); 0.5 wt % of N-tetradecyl-2-pyrrolidone (Sample H); 0.5 wt % of N-octadecyl-2-pyrrolidone (Sample I) and 0.5 wt % of N-methyl-2-pyrrolidone (Sample J) were substituted for 0.5 wt% of N-octyl-2-pyrrolidone. The dissolution rates for these pyrrolidones were measured, the results of which are reported in Table III.

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TABLE III

Sample	After 30 min	After 60 min	After 90 min	After 120 min
G	19.26	51.18	78.15	96.09
H	19.15	61.81	87.40	92.52
I	6.60	19.92	34.78	50.63
J	6.49	16.51	26.97	39.19
Control	7.93	20.80	33.46	44.80

The above results indicate that all of the pyrrolidones tested significantly increased the drug dissolution rate over that of the control; however, the results achieved with N-octyl pyrrolidone were outstanding and C₁₀ to C₁₄ alkyl pyrrolidones were only slightly less effective. The dissolution rate increasing effect falls off markedly with the C₁₈ alkyl pyrrolidone and is virtually non-existent in the lower molecular weight solvent type pyrrolidones which lack surfactant properties.

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EXAMPLE XLII

To improve the dissolution rate of chlorothiazide tablets, the following blends were made up:

			% by Wt.
5	K	Chlorothiazide	10.00
		N-dodecyl-2-pyrrolidone	0.50
		1:1 mixture of lactose:dicalcium phosphate	88.50
		Magnesium stearate	1.00
10	L	Chlorothiazide	10.00
		N-dodecyl-2-pyrrolidone	0.25
		1:1 mixture of lactose:dicalcium phosphate	88.75
		Magnesium stearate	1.00
15	M	Chlorothiazide	10.00
		N-dodecyl-2-pyrrolidone	0.10
		1:1 mixture of lactose:dicalcium phosphate	88.90
		Magnesium stearate	1.00
20	N	Chlorothiazide	10.00
		1:1 mixture of lactose:dicalcium phosphate	99.00
		Magnesium stearate	1.00

25 Blends K-M were prepared by mixing the chlorothiazide with an ethanolic solution of N-dodecyl-2-pyrrolidone and dried in an oven at 60°C. The dried material was blended with the lactose/dicalcium phosphate diluent for 7 minutes, after which the magnesium stearate was added and mixed for an additional 3 minutes. The

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resulting blends were then compressed into tablets of 8-10 Kp hardness and the dissolution of chlorothiazide was measured for each of K, L and M as reported in Table IV.

5 The blend of Sample N was similarly prepared except that N-dodecyl-2-pyrrolidone was omitted. This sample served as a control.

TABLE IV

	Sample	After 60 min.	After 120 min.	After 180 min.
10	K	49.14	90.24	99.26
	L	58.06	96.33	103.29
	M	13.48	23.81	56.54
	N	16.63	24.79	31.09

15 The above data indicates the excellent dissolution properties of N-dodecyl-2-pyrrolidone when employed in the above blends at a concentration of at least 0.25% by weight. Even at the 0.1 wt % concentration level, some improvement was noted after 3 hours.

EXAMPLE XLIII

.20 DEMONSTRATION OF SLIP AND ANTIBLOCK PROPERTIES

1,000 ppm of N-dodecyl-2-pyrrolidone was mixed into molten polypropylene having a melt flow index 8 and a density of 0.902 at 200°C. The resulting melt was conducted through an in-line mixer to an extruder from which a 1 mil blown
25 film was deposited on a clean stainless steel surface. The above procedure was repeated, except that the pyrrolidone was omitted. The coefficients of friction for the films

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were measured and the pyrrolidone containing film indicated a significant reduction compared to the untreated film. This reduction or "slip" development reaches a maximum within 24 hours, indicating rapid blooming of the pyrrolidone to the film surface. The following Table V summarized the data.

TABLE V

Coefficient of Friction: Cast Polypropylene Film

Elapsed Time Hours	Untreated Polypropylene	Film containing N-C ₁₂ -pyrrolidone (1,000 ppm)	Film containing Kemamide F (Erucamide) (1,000 ppm)
1	1.2	0.50	0.73
3	1.2	0.38	0.65
6	1.2	0.25	0.61
24	1.2	0.21	0.48
48	1.2	0.21	0.44

The data indicates the superior utility of the N-dodecyl pyrrolidone in the above melt.

EXAMPLE XLIVDemonstration of Dye Enhancing Properties

A low density polyethylene having a melt index 8 at 200°C. was mixed with 0.5% by weight N-dodecyl-2-pyrrolidone. The resulting uniform mixture was transferred to an extruder from which clothes-hangers were produced.

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The procedure was repeated except that mixture with a pyrrolidone was omitted. Unlike the untreated polymer, the N-docecyl-2-pyrrolidone containing thermoplastic absorbed 0.1% cationic dye having an anionic charge, (CI Acid Yellow 49), from boiling water upon immersion for one hour.

No absorption occurred with the pyrrolidone deficient melt. Other suitable acid anionic dyes which are beneficially absorbed by the lactams of this invention include CI Acid Blue 40, CI Acid Red 337, CI Acid Yellow 159, CI Acid Yellow 79 and CI Acid Yellow 151.

The following examples illustrates a low-energy process for removing organic pollutants from aqueous streams in waste water treatment. The waste waters included within the scope of this invention are sewage, wastes from industrial processes, such as for example a pulp paper mill, chemicals manufacture, plastic manufacturing, dairy and food processing; industrial spills of fuel oil and other contaminating chemicals and power plant waste waters. Of particular interest are the phenolic, halogen, mercapto, sulfur dioxide, nitric oxide and urea type contaminants contained in such waste waters.

Phenolic compounds are specially troublesome because, not only are they widely used in a great number of applications ranging from agricultural chemicals to food additives but also because they are unavoidably formed in many industrial processes. Phenolics are known to impart a disagreeable taste and odor to drinking water and edible aquatic life forms and are believed to be harmful against man and/or the environment. However, current methods for phenolic decontamination are time-consuming and laborious. The present low energy process provides an efficient method for removing these contaminants.

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EXAMPLE XLV

Aqueous 100 ml solutions of the following phenolics were prepared in 1 liter glass flasks. To these aqueous solutions was added N-octyl-2-pyrrolidone in the amounts indicated in Table VI. After the addition, the contents of the flasks were allowed to stand for 15 to 30 minutes, whereupon two liquid layers separated, i.e. an upper organic layer containing phenolics and N-octyl-2-pyrrolidone and a lower water layer. The layers were separated and weighed for phenolic content which was determined by UV absorption.

TABLE VI

	Sample	Grams of Phenolic in Water	% N-C ₈ P added to soln.	Grams of Phenolic in Separated Lactam Soln.	% Phenolic in Separated Lactam Soln.
15	1	0.02 g. Phenol	2	0.02	> 90
20	2	0.02 g. 2,4-dimethyl Phenol	2	0.02	> 90
25	3	0.03 g. Penta-chloro Phenol	2	0.03	> 90

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EXAMPLE XLVI

A 50 ml. deionized water sample containing an organophosphorous salt, namely a 1:1 ratio of dialkyl phosphate and tetraphenyl arsenic chloride salt was contacted with 4 ml. of N-octyl-2-pyrrolidone. After completion of the addition, the resulting mixture was allowed to stand for 15 minutes at room temperature; whereupon 2 layers separated: a lower water layer and an upper pyrrolidone layer. Analysis showed that the separated pyrrolidone layer contained more than 80% of the organophosphorous salt compound.

EXAMPLE XLVII

The procedure described in Example XLVI was repeated except that Bacteriophage virus was substituted for the organophosphorous salt. The separated upper N-octyl-2-pyrrolidone layer contained more than 70% of the virus.

EXAMPLE XLVIII

The procedure described in Example XLVI was repeated except that Escherichia coli was substituted for the organophosphorous salt and N-dodecyl-2-pyrrolidone was substituted for N-octyl-2-pyrrolidone. The separated upper N-dodecyl-2-pyrrolidone layer contained the Escherichia coli and none was found in the lower water layer.

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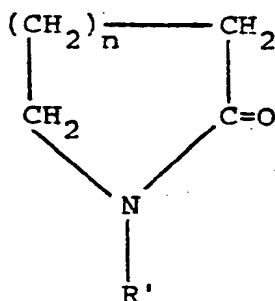
It is to be understood that the foregoing examples are merely representative and serve to illustrate the diverse properties discovered for the present lactams. Because of the multifarious effects of the present lactams, many more applications, modifications and alterations than specifically disclosed in the foregoing specification will become apparent to those skilled in the art. These are also within the scope of this invention. Particularly included are substitutions of any of the lactams disclosed herein in any of the above formulations and examples and any particular applications which derive from the unique properties of the present group of lactams including their complexability, surfactant properties, viscosity building, foam stabilizing, and surface tension reducing properties.

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SURFACE ACTIVE LACTAMS

WHAT IS CLAIMED IS:

1. A N-hydrocarbon substituted lactam surfactant having the formula



wherein n is an integer having a value of from 1 to 3 and R' is a hydrophobic radical consisting of a linear, branched chain or cyclic alkyl radical containing from 7 to 22 carbon atoms; a naphthyl or alkyl substituted naphthyl radical containing from 10 to 26 carbon atoms and an alkylphenyl or phenylalkyl radical containing from 9 to 26 carbon atoms; which lactams are capable of forming micelles in neutral, basic or acidic aqueous media and have a critical micelle concentration of between about 1×10^{-3} and about 5×10^{-5} moles per liter.

2. A composition comprising the compound of Claim 1 and an inert carrier therefor.

3. The compound of Claim 1 wherein R' is alkyl.

4. The compound of Claim 1 wherein n is 1 and R' is alkyl having from 8 to 18 carbon atoms.

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5. The composition of Claim 2 which additionally contains a compound capable of complexing with the compound of Claim 1 which is selected from the group consisting of compounds containing a phenolic group, compounds having an acidic hydrogen, compounds having a polarizable structure, a halogen, a metal oxide a mercaptan, a urea, a radioactive material and mixtures thereof.

6. The composition of Claim 5 wherein said composition contains a gaseous radioactive halogen.

7. The composition of Claim 5 wherein said composition is a disinfecting composition containing iodine, hydrogen iodide or an iodine salt.

8. The composition which comprises a compound of Claim 1, water and phenol or a phenol containing compound.

9. The composition of Claim 8 wherein said phenol containing compound is hydroquinone, vanillin or a halogenated phenol.

10. An emulsion concentrate which comprises a compound of Claim 1 and a water insoluble agricultural chemical.

11. The emulsion of Claim 10 wherein said agricultural chemical is selected from the group consisting of 2,4-dichlorophenoxyacetic acid, 2,4,6-trichlorophenol, 2,4,5,-trichlorophenoxyacetic acid, chlordane, chlorinated camphene, phenylmercuric acetate, phenyl mercuric naphthenate, phenyl mercuric oleate, aldrin, dieldrin, isopropyl phenylcarbamate and triazine.

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12. A paint and/or varnish removing composition containing an effective substrate penetrating amount of the compound of Claim 1.

13. A rodenticide composition containing an effective viscosity building amount of the compound of Claim 1.

14. An insect repellant composition containing an effective skin coating and skin protective amount of the compound of Claim 1.

15. An anti rust formulation containing an effective metal oxide complexing amount of the compound of Claim 1.

16. A perfume containing an effective odor retaining amount of the compound of Claim 1.

17. A spot and stain removing composition containing an effective promotional spot and stain removing amount of the compound of Claim 1.

18. A cosmetic nail treating composition containing an effective nail penetrating amount of the compound of Claim 1.

19. A beverage clarifying composition containing an effective phenolic complexing amount of the compound of Claim 1.

20. A medicinal pill, capsule or tablet containing an effective rate dissolution enhancing amount of the compound of Claim 1.

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21. A dye formulation containing an effective dye penetrating and dye exhausting amount of the compound of Claim 1.

22. A detergent composition containing an effective foam boosting amount of the compound of Claim 1.

23. A detergent composition containing an effective disinfecting amount of the compound of Claim 1 in the form of a complex with a disinfecting agent.

24. The detergent composition of Claim 23 wherein said composition is a liquid dairy cleaning composition.

25. The detergent composition of Claim 23 wherein said composition is a liquid animal washing solution.

26. A detergent composition containing an effective surfactant amount of the compound of Claim 1.

27. The detergent composition of Claim 26 wherein said composition formulation is a dishwashing liquid.

28. The detergent composition of Claim 26 wherein said detergent composition is a commercial bottle washing composition.

29. The detergent composition of Claim 26 wherein said detergent composition is a laundry composition.

30. The detergent composition of Claim 26 wherein said detergent composition is a liquid rug shampoo.

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31. The detergent composition of Claim 26 wherein said detergent composition is a liquid hard surface cleaner.

32. The detergent composition of Claim 26 wherein said detergent composition is a liquid cleaner for leather or plastics.

33. The composition of Claim 32 wherein the composition is a shoe polish.

34. The composition of Claim 26 wherein said detergent composition is a hard stone jewelry cleaning liquid.

35. A furniture polish containing an effective wood penetrating amount of the compound of Claim 1.

36. A laundry detergent composition containing an effective textile softening amount of the compound of Claim 1.

37. An aqueous rinsing liquid containing an effective detergent extracting amount of the compound of Claim 1.

38. A laundry detergent composition containing an effective antistatic amount of the compound of Claim 1.

39. An antistatic composition containing an effective antistatic amount of the compound of Claim 1 and an inert carrier therefor.

40. A complexable toxic chemical composition containing a toxic chemical and an effective complexing amount of the compound of Claim 1 which lowers the toxicity of said chemical.

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41. A normally liquid mineral acid containing an effective thickening amount of the compound of Claim 1.

42. A cosmetic composition containing an effective moisturizing amount of the compound of Claim 1.

43. The composition of Claim 42 wherein the cosmetic composition is selected from the group consisting of a face cream, a sunscreen, a shave cream, a hair shampoo, a body shampoo, a hand cream, a hair setting lotion, a foot lotion, a makeup foundation and a lipstick formulation.

44. A metal polish containing an effective oxide complexing amount of the compound of Claim 1.

45. An anti-block and slip agent suitable in the manufacture of molded plastics which comprises the compound of Claim 1 and a carrier therefor.

46. An anti-block and slip composition which comprises a moldable extrudable plastic and an effective anti-block and slip amount of the compound of Claim 1.

47. A highly toxic chemical compound composition containing an effective toxic reducing and complexing amount of the compound of Claim 1.

48. The composition of Claim 47 wherein said toxic chemical compound is selected from the group of aldrin, dieldrin and a phenyl mercuric compound.

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49. The composition of Claim 48 where said toxic chemical compound is a phenyl mercuric compound selected from the group consisting of the phenyl mercuric acetate, borate, chloride, hydroxide, naphthenate, nitrate, oleate, propionate and salicylate.

50. The process which comprises adding an effective complexing amount of the compound of Claim 1 to a complexible compound selected from the group consisting of a phenol, a compound having an acidic hydrogen, a compound having a polarizable structure, a halogen, a metal oxide, a mercaptan, a urea, and a radioactive halogen.

51. The process which comprises contacting a metal surface in an acidic environment with an effective corrosion inhibiting amount of the compound of Claim 1.

52. The process which comprises adding to a detergent composition at a temperature conducive to foaming, a foam enhancing and foam stabilizing amount of the compound of Claim 1.

53. The process of Claim 52 wherein the compound of Claim 1 is an N-alkyl pyrrolidone having from 8 to 18 carbon atoms in the alkyl group.

54. The process which comprises adding during a washing and/or drying cycle an effective static charge depressing amount of the compound of Claim 1 to a fabric which is subjected to laundering and drying.

55. The process which comprises adding an effective moisturizing amount of the compound of Claim 1 to a cosmetic formulation.

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56. The process which comprises applying an effective complexing and skin penetrating amount of the compound of Claim 1 contained in a cosmetic composition, to an epidermal substrate.

57. The process which comprises adding an effective solubilizing amount of the compound of Claim 1 to a water insoluble compound.

58. The process of Claim 57 wherein the water insoluble compound is a drug.

59. The process which comprises adding an effective lubricating amount of the compound of Claim 1 to a slip and anti-block composition and applying the resulting composition to a surface subjected to friction.

60. The process which comprises adding an effective emulsifying amount of the compound of Claim 1 to a water insoluble compound to form a cream or paste composition.

61. The process which comprises adding an effective co-surfactant acting amount of the compound of Claim 1 to anionic surfactant.

62. The process of Claim 61 wherein the anionic surfactant is selected from the group consisting of an alkali metal lauryl sulfate, an alkali metal laureth sulfate, ammonium lauryl sulfate, triethanolamine lauryl sulfate and an α -olefin sulfonate.

63. The process which comprises adding an effective viscosity building amount of the compound of Claim 1 to a liquid composition to form a gel, cream or paste.

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64. The process which comprises applying an effective paint or resin stripping amount of the compound of Claim 1 to a paint or resin coated substrate.

65. The process which comprises adding an effective soil releasing amount of the compound of Claim 1 to a laundry detergent composition.

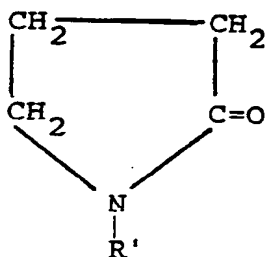
66. The process which comprises complexing the compound of Claim 1 with a water insoluble disinfectant solution and applying said complex to a surface for cleaning.

67. The process of Claim 66 wherein the compound of Claim 1 is complexed with iodine and the resulting complex added to a disinfectant solution.

68. The process which comprises applying the product of Claim 1 complexed with iodine in the form of a gel, over a wound as a barrier to bacterial infection.

69. The process which comprises adding an effective complexing and time releasing amount of the compound of Claim 1 to a drug or antiseptic formulation.

70. A method which comprises adding to an aqueous solution of a liquid having a surface tension of at least 35 dynes/cm, an effective surface tension reducing amount of the compound having the formula



wherein R' is alkyl having from 8 to 14 carbon atoms.

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71. The process which comprises adding effective clarifying amount of the compound of Claim 1 to a clouded beverage containing phenolic impurities.

72. The process of Claim 71 wherein said beverage is beer or wine.

73. The process which comprises adding an effective detergent promoting amount of the compound of Claim 1 to a standard detergent formulation.

74. The process of Claim 73 wherein the detergent formulation is selected from the group consisting of a dishwashing formulation, a laundry detergent formulation, a bottle washing formulation, a glass cleaning formulation, a leather cleaning formulation, a plastic cleaning formulation, a household cleaning formulation and a rug shampoo formulation.

75. The process which comprises adding an effective foam boosting amount of the compound of Claim 1 to a detergent formulation.

76. The process which comprises adding an effective sanitizing and disinfecting amount of the compound of Claim 1 to a sanitizing and disinfecting formulation.

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77. The process which comprises adding an effective detergent extracting amount of the compound of Claim 1 to the rinse water in a laundering operation.

78. The process which comprises adding an effective antistatic amount of the compound of Claim 1 to a textile, plastic or fiber.

79. The process of Claim 78 wherein said substrate is a textile.

80. The process of Claim 78 wherein said substrate is a rug.

81. The process of Claim 79 wherein said textile is clothing.

82. The process of Claim 78 wherein said substrate is a plastic and wherein said plastic is subsequently subjected to molding operation.

83. The process which comprises adding an effective skin softening amount of the compound of Claim 1 to a cosmetic formulation.

84. The process of Claim 83 wherein said cosmetic formulation is selected from the group consisting of a skin lotion, a facial cream, a sunscreen lotion, a hair shampoo, hair setting lotion, a body shampoo, a shaving cream, a hand lotion, a hand cleaner and a hair mousse.

85. The process which comprises adding an effective pesticidal promoting amount of the compound of Claim 1 to a pesticidal formulation.

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86. The process of Claim 85 wherein said pesticidal formulation is an antirodent formulation.

87. The process which comprises adding a effective promotional rust removing amount of the compound of Claim 1 to a rust removing formulation.

88. The process which comprises adding an effective skin penetrating and odor retaining amount of the compound of Claim 1 to a perfume.

89. The process which comprises contacting a woven or knitted substrate with an effective anti-soil depositing amount of the compound of Claim 1.

90. The process of Claim 89 wherein said substrate is the nap of a rug.

91. The process of Claim 89 wherein said substrate is an article of clothing.

92. The process which comprises adding an effective foam stabilizing amount of the compound of Claim 1 to a foam producing detergent formulation.

93. The process of contacting a radioactive halogen gas with an effective complexing amount of the compound of Claim 1 in aqueous solution.

94. The process of Claim 93 wherein the complexed radioactive gas in aqueous solution is employed as feed to a thermo fixation process.

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95. The process of washing a radioactive contaminated surface with a composition containing an effective complexing amount of the compound of Claim 1 in aqueous solution, an anionic wetting agent and a fatty amido betaine.

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96. The process which comprises washing vegetation contaminated by exposure to a radioactive material with an effective complexing amount of the compound of Claim 1 in aqueous solution.

97. The process which comprises washing equipment contaminated by exposure to a radioactive material with an effective complexing amount of the compound of Claim 1 in aqueous solution.

98. The process which comprises applying an effective textile penetrating, spot or stain removing amount of the compound of Claim 1 to a dry cleaning formulation.

99. The process which comprises applying an effective spot or stain removing amount of the compound of Claim 1 to the stained area of a woven substrate.

100. The process of Claim 99 wherein said substrate is an article of clothing.

101. The process of Claim 99 wherein said substrate is a rug.

102. The process of increasing the dissolution rate of an orally administered drug in the form of a tablet or capsule by incorporating in the pill or tablet formulation an effective 0.1 to 1 % amount of the compound of Claim 1.

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103. The process of Claim 102 wherein the drug is a chlorothiazide.

104. The process which comprises contacting an odor causing chemical selected from the group consisting of a mercaptan, an odoriferous tar, nicotine, urea and mold with an effective complexing amount of the compound of Claim 1.

105. The process of strengthening fingernails which comprises applying to the nail surface and surrounding cuticle area a nail building composition containing an effective cuticle penetrating and moisturizing amount of the compound of Claim 1.

106. The process of Claim 105 wherein said application is continued for a period of at least 10 days.

107. The process which comprises applying to an oxidized metal surface an effective oxide complexing amount of the compound of Claim 1.

108. The process of Claim 107 wherein the compound of Claim 1 is incorporated into a metal polish composition and the composition is applied to the metal surface.

109. The process which comprises applying to a wood surface a wood cleaning and polishing composition containing an effective pore penetrating and surfactant amount of the compound of Claim 1.

110. The process which comprises applying to a leather surface a leather cleaning composition containing an effective pore penetrating and surfactant amount of the compound of Claim 1.

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111. The process of Claim 110 wherein said leather cleaning composition is a shoe polish.

112. The process of Claim 110 wherein said leather cleaning composition is a leather furniture polish.

113. The process of Claim 50 wherein the complexable compound contacted with the compound of Claim 1 is contained in waste water.

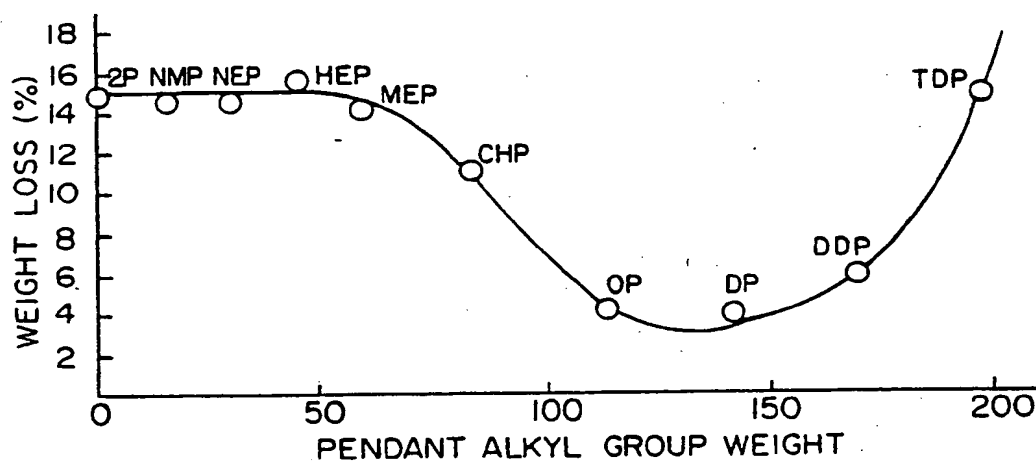


FIG. 1

INTERNATIONAL SEARCH REPORT

International Application No PCT/US87/01391

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) ³		
According to International Patent Classification (IPC) or to both National Classification and IPC		
IPC ⁴ CO7D 207/267, 27; 211/76; 223/10		
U.S. Cl. 540/485; 546/243; 548/543		
II. FIELDS SEARCHED		
Minimum Documentation Searched ⁴		
Classification System	Classification Symbols	
U.S.	540/485, 486, 531; 546/8, 9, 216, 243; 548/279, 402, 543; 134/38; 252/89.1, 106, 252/107, 175, 626, 387, 388, 542, Dig. 5, Dig. 6, Dig. 8, Dig. 14, Dig. 15, Dig. 19, 106/3, 14.16; 424/ Dig. 8, Dig. 10, 1.1, 401, 405, 406, 59, 60, 64, 70, 73; 524/910	
Documentation Searched other than Minimum Documentation to the extent that such Documents are Included in the Fields Searched ⁶		
US PATENT INDEX; US CHEMICAL PATENTS FILE 323 (1940-1970) DIALOG WORLD PATENT INDEX: CHEM. ABS. (STRUCTURE & WORD SEARCH)		
III. DOCUMENTS CONSIDERED TO BE RELEVANT ¹⁴		
Category ⁵	Citation of Document, ¹⁶ with indication, where appropriate, of the relevant passages ¹⁷	Relevant to Claim No. ¹⁸
X	JOURN. OF THE AMER. CHEM. SOC., VOLUME 80 MARCH 1947, F.B. ZIENTY AND G.W. STEAHLY, "N-SUBSTITUTED 2-PYRROLIDONES", PP. 715-717 (SEE PAGES 715-716).	1
X Y	CHEM. ABSTRACTS, VOL. 80, NO. 8, 41385Z; ISSUED 1974 FEBRUARY 24 (COLUMBUS, OHIO, U.S.A.), MASAYUKI ET AL., "PHASE SEPARATION AND SURFACE TENSION OF AQUEOUS HYDROCHLORIC ACID SOLUTION OF N-DODECYL-2-PYRROLIDONE, P. 298, COLUMNS 1 AND 2 NIPPON KAGAKU KAISHI, 1973, (11), 2056-62 (JAPAN).	1-4 5, 50
X	CHEM. ABSTRACTS, VOL. 84, NO. 26, 185540B, ISSUED 1976, JUNE 28 (COLUMBUS, OHIO U.S.A.) MASAYUKI ET AL., "THE EFFECT OF ADDITIVES ON THE SOLUBILITY OF 1-DODECYL-2- PYRROLIDONE IN AQUEOUS SOLUTIONS", P. 357, COL. 2, NIPPON KAGAKU KAISHI, 1976, (4), 547-53 (JAPAN).	1-4
<p>¹⁵ Special categories of cited documents:</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>"&" document member of the same patent family</p>		
IV. CERTIFICATION		
Date of the Actual Completion of the International Search ²		Date of Mailing of this International Search Report ²
28 SEPTEMBER 1987		04 NOV 1987
International Searching Authority ¹		Signature of Authorized Officer ²⁰
US PATENT & TRADEMARK OFFICE		ANGELA L. FUGO

Form PCT/ISA/210 (second sheet) (May 1986)

FURTHER INFORMATION CONTINUED FROM THE SECOND SHEET

V. ☐ OBSERVATIONS WHERE CERTAIN CLAIMS WERE FOUND UNSEARCHABLE ¹⁰

This international search report has not been established in respect of certain claims under Article 17(2) (a) for the following reasons:

1. ☐ Claim numbers because they relate to subject matter ¹² not required to be searched by this Authority, namely:

2. ☐ Claim numbers because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out ¹³, specifically:

VI. ☒ OBSERVATIONS WHERE UNITY OF INVENTION IS LACKING ¹¹

This International Searching Authority found multiple inventions in this international application as follows:

Group I: Claims 1-4

Group II: Claims 5-9, 19, 47-50, 67, 68, 71, 72

Group III: Claims 10 and 11

Group IV: Claims 12 and 64. (See attachment)

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims of the international application.
2. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims of the international application for which fees were paid, specifically claims:
3. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claim numbers:
4. ☐ As all searchable claims could be searched without effort justifying an additional fee, the International Searching Authority did not invite payment of any additional fee.

Remark on Protest

- ☐ The additional search fees were accompanied by applicant's protest.
- ☒ No protest accompanied the payment of additional search fees.

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Group V : Claims 13, 63, 85 and 86
Group VI : Claim 14
Group VII : Claims 15, 51 and 87
Group VIII : Claims 16 and 88
Group IX : Claims 17, 98-101
Group X : Claims 18, 105 and 106
Group XI : Claims 20, 57, 58, 102 and 103
Group XII : Claim 21
Group XIII : Claims 22-29, 30-32, 34, 36-38, 52, 53, 66, 73-76
92 and 110
Group XIV : Claims 33, 35, 109, 111 and 112
Group XV : Claims 39, 54 and 78-82
Group XVI : Claims 40 and 104
Group XVII : Claim 41
Group XVIII : Claims 42, 43, 55, 56, 83 and 84
Group XIX : Claims 44, 107 and 108
Group XX : Claims 45, 46 and 59
Group XXI : Claims 60-62
Group XXII : Claim 70
Group XXIII : Claims 89-91

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)

Category *	Citation of Document, with indication, where appropriate, of the relevant passages ¹⁷	Relevant to Claim No ¹⁸
X Y	CHEM. ABSTRACTS, VOL. 104, NO. 10, 74916r, ISSUED 1986, MARCH 10 (COLUMBUS, OHIO, USA) OHSHIMA ET AL., "ENHANCING EFFECT OF ABSORP- TION PROMOTERS... STUDY ON THE ABSORPTION PROMOTING EFFECT OF AZONE", P. 385, COL. 1, J. PHARMACOBIO-DYN., 1985, 8(11), 900-5(ENG).	1-4 20, 57, 58, 102 & 103
X Y	CHEM. ABSTRACTS, VOL. 105, NO. 18, 158716j, ISSUED 1986, NOVEMBER 3 (COLUMBUS, OHIO, USA) MARAKAMI ET AL., "PROMOTING EFFECT OF AZONE ON INTESTINAL ABSORPTION OF POORLY ABSORBABLE DRUGS IN RATS", P. 366, COL. 2, INT. J. PHARM., 1986, 31(3), 231-8(ENG.)	1-4 20, 57, 58, 102 & 103
Y	CHEM. ABSTRACTS, VOL. 87, NO. 23, 184365y, ISSUED 1977, DECEMBER 5 (COLUMBUS, OHIO, USA) SIMAK ET AL., "IODINE COMPLEXES OF DIALKYL ACID AMIDES AND N-ALKYL LACTAMS", P. 598, COL. 2, GER OFFEN. 2611602 (Cl.CO7D 207/26) 22 SEP. 1977, 14PP.	5-7, 50, 66-68 93, 94, 113
Y	CHEM. ABSTRACTS, VOL. 91, NO. 23, 192602W; ISSUED 1979, DECEMBER 3 (COLUMBUS, OHIO USA) RUOSTESUO, "STUDIES ON THE POLARITY OF 1-PHENYL-2-PYRROLIDINONES, SULFONAMIDES AND SULFENAMIDES", P.582, COLUMNS 1 AND 2, ACTA UNIV. OULUENSIS, SER. A 1978, 66, 53 PP. (ENG.)	5-7, 50, 66-68 93, 94, 113
Y	CHEM. ABSTRACTS, VOL. 95, NO. 26, 230648h ISSUED 1981, DECEMBER 28 (COLUMBUS, OHIO USA) JPN. TOKKOYO KOHO 81 35,018 (Cl. HO1G9/02, 14 AUG. 1981, NICHICON CAPACITOR LTD., 5PP.	5, 8 AND 50
X	CHEM. ABSTRACTS, VOL. 90, NO. 25, 203295f, ISSUED 1979, JUNE 18 (COLUMBUS, OHIO, USA) KARJALAINEN, ET AL.. "HYDROGEN BONDING OF PYRROLE, INDOLE AND CARBAZOLE WITH SUB- STITUTED 1-PHENYL-2-PYRROLIDINONES", P.544, COLUMNS 1 AND 2, ACTA CHEM. SCANDA., SER. A 1979, A33(1), 76-8 (ENG.)	5 AND 50
Y	CHEM. ABSTRACTS, VOL. 93, NO. 25, 232708K, ISSUED 1980, DECEMBER 22 (COLUMBUS, OHIO USA) MCCARTHY ET AL. "MIXTURES OR ADDITION COMPOUNDS OF HALOALKYL PHOSPHONIC ACIDS AND AMIDES OR AMIDE POLYMERS, AND THEIR USE AS PLANT GROWTH REGULANTS", P.247, COL. 1, BRIT. UK PAT. APPL. 2029832 (Cl.CO7F9/28), 26 MAR. 1980, US APPL. 935945, 23 AUG. 1978; 24PP.	5, 10 AND 50

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)		
Category *	Citation of Document, ¹⁶ with indication, where appropriate, of the relevant passages ¹⁷	Relevant to Claim No ¹⁸
Y	CHEM. ABSTRACTS, VOL. 95, NO. 21, 187417t ISSUED 1981, NOVEMBER 23, (COLUMBUS, OHIO USA) MCCARTHY ET AL (GAF CORP.) FR. DEMANDE 2447930 (Cl. CO7F9/65), 29 AUG. 1980, US APPL. 7769, 30 JAN. 1979; PHOSPHORANYL DERIVATIVES CONTAINING POSITIVELY CHARGED NITROGEN", 36 PP.	5, 10 and 50
X	CHEM. ABSTRACTS, VOL. 78, NO. 1 75P. ISSUED 1973, JANUARY 8 (COLUMBUS, OHIO, USA) YOSHIHARA ET AL., "METABOLISM OF DRUGS. LXXVI. METABOLIC FATE OF PROLINTANE IN RABBITS" P.83, COL. 1, CHEM. PHARM. BULL. 1972, 20(9), 1906-12 (ENG.)	5 AND 50
X	CHEM. ABSTRACTS, VOL. 84, NO. 19, 134762t, ISSUED 1976, MAY 10 COLUMBUS, OHIO, USA) VIRTANEN ET AL., "THE ASSOCIATION OF SUB- STITUTED 1-PHENYL-2-PYRROLIDONES WITH PHENOL", P.425, COL. 1, FINN. CHEM. LETT. 1975, (7-8), 183-6 (ENG.)	1-5, 8, 9, 50
Y	CHEM. ABSTRACTS, VOL. 98, NO. 15, 125489w, ISSUED 1983, APRIL 11 (COLUMBUS, OHIO, USA) YAMAMOTO ET AL." "ULLMAN CONDENSATION USING COPPER OR COPPER OXIDE AS THE REACTANT. ARYLATION OF ACTIVE HYDROGEN COMPOUNDS (IMIDES, AMIDES, AMINES, PHENOL, BENZOIC ACID AND PHENYLACETYLENE", P.586, COL. 1, CAN. J. CHEM. 1983 61(1), 86-91 (ENG.).	5, 44, 50, 107
X	US, A 4,557,934 PUBLISHED 10 DECEMBER 1985, (COOPER)	1-4
$\frac{X}{Y}$	US, A, 3,988,318 PUBLISHED 26 OCTOBER 1976, (COPESE ET AL)	$\frac{1-5}{6-113}$
$\frac{X}{Y}$	US, A, 3,988,350, PUBLISHED 26 OCTOBER 1976	$\frac{1-5}{6-113}$

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- Group II: - Claims 5-9, 19, 47-50, 67, 68, 71, 72 complexing compositions and various methods of forming the complexing compositions with the compounds cited in claim 5.
- Group III: Claims 10 and 11, an emulsion concentrate composition.
- Group IV: Claims 12 and 64, a paint removing composition and method of using the composition.
- Group V: Claims 13, 63, 85 and 86, a pesticide/rodenticide composition and methods for making such compositions.

- Group VI: Claim 14, an insect repellant composition.
- Group VII: Claims 15, 51 and 87, a rust removing composition and methods of making and/or using the composition.
- Group VIII: Claims 16 and 88, a perfume and method of making the same.
- Group IX: Claims 17, 98-101, a spot and stain removing composition and method of using.
- Group X: Claims 18, 105 and 106, a nail.
- Group XI: Claims 20, 57, 58, 102 and 103, drug compositions and method of making.
- Group XII: Claim 21, a dye formation.

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- Group XIII: Claims 22-29, 30-32, 34, 36-38, 52, 52, 53, 66, 69, 73-76, 92 and 110 are detergent compositions and various methods of making them.
- Group XIV: Claims 33, 35, 109, 111 and 112, polishes methods of using them.
- Group XV: Claims 39, 54 and 78-82, antistatic compositions and methods for their use.
- Group XVI: Claims 40, and 104, complexible toxic chemical compositions and methods for using the same.
- Group XVII: Claim 41, a mineral acid composition.
- Group XVIII: Claims 42, 43, 55, 56, 83 and 84, cosmetic composition and method of making

- and/or using such compositions.
- Group XIX: Claims 44, 107, and 108, metal polishing compositions and method of making and/or using the compositions.
- Group XX: Claims 45, 46 and 59, an anti-block and slip composition and method of making or using the composition.
- Group XXI: Claims 60-62, the process of emulsifying with the claimed compound.
- Group XXII: Claim 70, a method of reducing the surface tension of a liquid.
- Group XXIII: Claims 89-91, a process of coating with an anti-soil composition.

The complex of claim 5 is a completely different structure than the compound of claims 1, 3 and 4. Hence, Groups I and IV are mutually exclusive species.

Additional groups provide various compositions and method of making and/or using the compositions. All of these groups are mutually exclusive of each other in that they normally contain different ingredients. In addition, they are classified and searched in separate areas.

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